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Issue of Journal. The Journal appears in four parts per sessional volume, viz., in January, April, July and October. A brochure describing the origin, progress and purposes of the Institution, and comprising also the Memorandum and Articles of Association, the By-Laws and Regulations of the Institution, the Library Catalogue to date (with subject index), and the List of Members, was published in September, 1915, to be followed periodically by revisions of the Library Catalogue and List of Members. The List of Members brought up to March, 1922, is now issued.

Members of all classes are entitled to receive these publications free; for additional copies of the Journal they will be charged at the price of seven shillings and sixpence per part, and of the brochures at the prices stated on the wrappers, varying in proportion to bulk.

Changes of Address. It is particularly requested that members notify the Secretary immediately of any change of address; and members are also requested to advise the Parcel Post Department, as well as the Letter Office, of any temporary change of address, as, unless this is done, parcel post packets will not be re-addressed, but will be returned to the offices of the Institution in London, thus incurring further expense for postage.

To Authors of Papers. Papers should be written in the third person and the copy should be carefully corrected by the author before it is presented.

All drawings, diagrams or other illustrations should be sent in a fit state for direct photographic reproduction.

All quotations, technical terms and localisms should be indicated by means of inverted commas.

Foreign weights, measures and costs should be given whenever possible, and also their English equivalents.

It is suggested that authors append as complete a bibliography of the subject treated as may be possible.

All papers and notes submitted to the Institution are supposed to be original communications unless distinctly stated to be otherwise, in which case the exact reference to the previous publication should be given.

Communications upon papers read at meetings, notices of personal movements, or other matter for which publication is desired in the next issue of the Journal, should be in the hands of the Editor on or before the last day of December, March, June or September, as the case may be. Subsequent delivery may be too late for insertion.

The Council desire to draw the attention of authors to the serious increase in the cost of production. Papers, both for the Journal and for communication to the Institution, should be as concise and condensed as possible.

Communications. The Council invite members to submit papers for the forthcoming session. Communications to the Institution will, subject to the approval of the Publication Committee, be published in the Journal.

Appointments Register. A Register will be kept at the offices of the Institution for the convenience of firms requiring the services of members and for members requiring appointments, but on the distinct understanding that the Institution accepts no responsibility and gives no guarantee.

Library. The Institution's Library at No. 5 John Street, Adelphi, may be consulted between 11 and 4 daily. Extensive additions are being made to the current and standard literature on Petroleum.

Additions since publication of last Journal :—

Proceedings of the Institution of Automobile Engineers, 1921–22. Part I.

Journal of the Iron and Steel Institute. Vol. CV., No. 1, 1922.

Proceedings of the Institution of Mechanical Engineers. Vol. 1, 1922.

Proceedings of the Institution of Civil Engineers. Vol. CCXII., 1920–21. Part 2.

Theory of Wave Transmission. A Treatise on Transmission of Power by Vibrations. By George Constantinesco.

Sedimentary Petrography. By Henry B. Milner.

The Chemical Composition of the Esthonian M.-Ordovician Oil-Bearing Mineral, Kukkersite. By P. N. Kogerman.

Handbook of the Petroleum Industry. Vols. I. and II. By Dr. David T. Day.

The Petroleum and Allied Industries. By James Kewley.

The Marine Steam Engine. By R. Sennett and H. J. Oram.

Some Experiments in Connection with the Injection and Combustion of Fuel Oil in Diesel Engines, and Discussions, Correspondence, and Author's Reply. By Commander C. J. Hawkes, R.N.

Technology of Petroleum. By Henri Neuburger and Henri Noalhat.

Studiul Petrolului Romin. By Dr. L. Edeleanu and I. Tanesescu.

From the Imperial Mineral Resources Bureau :—

Iron Ore (Summary of Information as to the Present and Prospective Iron-Ore Supplies of the World). Parts II., British Africa; III., British America; IV., British Asia; V., Australia and New Zealand; VII., Foreign America; and VIII., Foreign Asia.

The Mineral Industry of the British Empire and Foreign Countries. War Period (1913–1919). Quicksilver. Vanadium.

From the Department of the Interior, U.S. Geological Survey :—

Bulletin No. 192. Carbon Black. Its Manufacture, Properties, and Uses. By R. O. Neal and G. St. J. Perrott.

Bulletin No. 730B. Erosion and Sedimentation in the Papago Country, Arizona. With a Sketch of the Geology. By Kirk Bryan.

Bulletin No. 735D. Silver Enrichment in the San Juan Mountains, Colorado. By Edson S. Bastin.

Bulletin No. 736A. The Structure of the Madill-Denison Area, Oklahoma and Texas. With Notes on Oil and Gas Development. By O. B. Hopkins, Sidney Powers, and H. M. Robinson.

Bulletin No. 736C. Geology of the Wiles Area, Ranger District, Texas. By Carroll E. Dobbin.

Professional Paper No. 129G. The Flora of the Woodbine Sand at Arthurs Bluff, Texas. By Edward Wilber Berry.

Professional Paper No. 129H. Geology of the Lower Gila Region, Arizona. By Clyde P. Ross.

Professional Paper No. 129I. The Flora of the Cheyenne Sandstone of Kansas. By Edward Wilber Berry.

Water-Supply Paper No. 477. Surface Water Supply of the United States, 1918. Part VII. Lower Mississippi River Basin. By Nathan C. Grover and Others.

Water-Supply Paper No. 487. The Arkansas River Flood of June 3–5, 1921. By Robert Follansbee and Edward E. Jones.

Technical Paper No. 263. Design and Operation of a Low-Pressure Absorption Plant. By W. P. Dykema and A. A. Chenoweth.

Technical Paper No. 298. Methods of Testing Petroleum Products.

Production Technology and Uses of Petroleum and Its Products, etc.

Copper in 1920. By H. A. C. Jenison.

Natural-Gas Gasoline in 1920. By E. G. Sievers.

Gold, Silver, Copper, Lead, and Zinc in New Mexico and Texas in 1920. By Charles W. Henderson.

Sulphur, Pyrites, and Sulphuric Acid in 1920. By Philip S. Smith.

Gold and Silver in 1920. By J. P. Dunlop.

Gold, Silver, Copper, Lead, and Zinc in Colorado in 1920. By Charles W. Henderson.

Asbestos in 1920. By Edward Sampson.

Fuel Briquets in 1921. By W. F. McKenney.

Chromite in 1921. By Edward Sampson.

Gold, Silver, Copper, Lead, and Zinc in the Eastern States in 1921. By J. P. Dunlop.

Abrasive Materials in 1921. By L. M. Beach and A. T. Coons.

Magnesium in 1921. By G. F. Loughlin.

Silica in 1921. By L. M. Beach.

Fuller's Earth in 1921. By Jefferson Middleton.

Peat in 1921. By K. W. Cottrell.

Graphite in 1921. By L. M. Beach.

Zinc in 1921. By C. E. Siebenthal and A. Stoll.

Lead in 1921. By C. E. Siebenthal and A. Stoll.

Manganese and Manganiferous Ores in 1921. By H. A. C. Jenison.

Lead and Zinc Pigments and Salts in 1921. By C. E. Siebenthal and A. Stoll

Slate in 1921. By G. F. Loughlin and A. T. Coons.

Strontium in 1921. By George W. Stose.

Carbon Black Produced from Natural Gas in 1921. By E. G. Sievers.

Fluorspar and Cryolite in 1921. By Herbert W. Davis.

Potash in 1921. By M. R. Nourse.

Phosphate Rock in 1921. By K. W. Cottrell.

Cadmium in 1921.

Mineral Resources of the United States, 1917. By H. D. McCaskey. **Part II. Non-Metals.** By G. F. Loughlin.

From the Department of Mines, Mines Branch, Canada :—

Barium and Strontium in Canada. By Hugh S. Spence.

Summary Report, 1921. Part C. Geology and Mineral Resources of Rice Lake and Oiseau River Areas, Manitoba. By H. C. Cooke.

Bulletin No. 34. Geological Series No. 41. Physiography and Glacial Geology of Gaspe Peninsula, Quebec. By A. P. Coleman.

From the Industrial Research Department, University of Alberta :—

Second Annual Report of the Scientific and Industrial Research Council of Alberta, Report No. 5.

From the Mines and Quarries Department, Ministry of Finance, Egypt :—

Report on the Mineral Industry of Egypt.

From the British Engineering Standards Association :—

Complete Sets of Publications and Specifications.

The attention of members is drawn to the new supplement of the Journal which deals with current Petroleum literature. To make this section of the maximum usefulness it is necessary that there should be full co-operation between chemists, engineers and geologists, and that matter which has inadvertently escaped notice shall be immediately sent to the Editor. The Institution is indebted to Mr. W. J. Wilson for the compilation of the bibliography.

Advertisements in the Journal. A limited number of Advertisements of firms interested in the Petroleum Industry may be inserted in the Journal. Application for terms, etc., should be made to Messrs. Commercial Publicity Service, 58, Warwick Street, Regent Street, London, W.1.

LIST OF ADVERTISERS.

(Members are desired, when making enquiries or placing orders with advertisers, to mention that they have seen their announcement in the Journal.)

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PERSONAL NOTES OF MEMBERS AND SPECIAL NOTICES.

It is suggested that Members of the Institution send information regarding their movements to the Secretary for insertion under this heading.

MR. H. TATHAM has joined the staff of the British Burma Petroleum Co., Ltd., and has sailed for Burma.

MR. GEORGE P. CHAPLIN has left England for Burma.

MR. H. LESLIE LITTLE has left for Barbados.

DR. J. A. L. HENDERSON has sailed for Canada and the U.S.A and expects to be away until about Christmas.

MR. W. SUTTON left England early in October for a short visit to Egypt.

MR. R. R. THOMPSON has resigned his position as Director of Lands and Mines of Trinidad on his appointment to the Professorship of Oil Mining at the Birmingham University.

The Council regret to announce that they have just received intimation of the death of a member, namely, Mr. T. H. VAUGHAN, of Redlands, California.

THE LATE MR. GEORGE TWEEDY.

It is with great regret that we have to record the death of Mr. George Tweedy, one of our Founder Members, who passed away at Eastbourne on August 20th.

Mr. Tweedy, who was 72 years of age, was born at Ulgham, in Northumberland. He has been closely connected with the Russian Petroleum Industry for over thirty years, and was Chairman and Managing Director of a number of Russian companies. During his life he made numerous journeys to the Russian oilfields, and was instrumental in bringing about many reforms in the conditions of the working classes of that country.

Mr. Tweedy, who will be greatly missed by his many associates and friends, leaves three daughters, and to them we tender our sincerest sympathy in their great loss.

ABBREVIATIONS.

Members are reminded that the correct abbreviations for use to indicate membership of the Institution are :—

Honorary Members ..	Hon.M.Inst.P.T.
Members	M.Inst.P.T.
Associate Members ..	A.M.Inst.P.T.
Students	Stud.Inst.P.T.
Associates	Assoc.Inst.P.T.

THE SIR JOHN CASS TECHNICAL INSTITUTE.

The classes in the Department of Petroleum Technology of this Institute will commence on October 9th, and we note that the syllabus of the First Year's Course includes the following special lectures by members of our Institution :—

"The Origin of Petroleum." By T. Dewhurst.

"Typical Oil Field Conditions." By W. R. A. Weatherhead.

"Application of Petroleum Products as Fuels." By Prof. J. S. S. Brame.

"Application of Petroleum Products—Illumination, Lubrication, etc." By Dr. A. E. Dunstan.

"The Natural Gas Industry." By James Kewley.

A lecture on the Shale Oil Industry will also be given by Mr. E. M. Bailey.

* * * *

A copy of the complete syllabus of the classes can be obtained from The Sir John Cass Technical Institute, Jewry Street, Aldgate, London, E.C. 3.

CHELSEA POLYTECHNIC.

We note that a course of thirty-six lectures, followed by practical work, on "Chemistry and Technology of Petroleum" is being given at this Institute by one of our Members, Mr. Fred Esling, commencing on September 28th. Those desiring further information should apply to the Head of the Chemical Department, Chelsea Polytechnic, Manresa Road, London, S.W. 3.

The Shale Oil Industry of Scotland.

By Edwin M. Bailey.

The Oil-Shale which is the raw material of the Shale Oil Industry is a mineral obtained by mining, usually at a considerable depth. It consists of hardened clay, rich in what was at one time animal and vegetable remains, and contains only traces of oily material extractable by solvents, but contains certain organic material or "kerogen," which, under certain conditions which it is the aim of the manufacturer to secure, yields liquid oil and wax. When the shale is heated to redness in retorts, in the absence of air but in the presence of steam, the elements (chiefly carbon and hydrogen, with a little oxygen, nitrogen and sulphur) of which this organic matter consists re-arrange themselves in a large number of new forms, the result being a wonderful variety of different oils and other substances. A simple analogy may be found in the building up of an oratorio from eight musical notes, or a great picture from a few primary colours. These products have a wide range of specific gravity, boiling-point and melting-point, and possess varied and valuable properties. From the mine-head the shale is conveyed in trucks by an endless wire rope to the breaker, where it is broken into pieces of suitable size (about 4-inch cube) by passing between revolving heavy toothed C.I. rollers fitted with wedge-shaped blunt teeth made of specially tempered hard steel, and conveyed by trucks and endless chain up a sloping scaffold to the top of the bench of retorts, which is usually composed of 60 single retorts. There are several types of retort, and one of these may be briefly described. It is vertical, and circular in section. At the top there is a M.S. hopper which feeds the shale into the retort, the upper part of which consists of C.I., and is 11 feet 3 inches long and 2 feet diameter at the top, tapering outwards to 2 feet 4 inches at the bottom. The lower part of the retort is constructed of fire-brick, and is 22 feet 18 inches long, and 2 feet 4 inches diameter at the top, tapering outwards to 3 feet at the bottom. The retorts are arranged in sets of four within heating chambers in the bench, and each pair of retorts has hoppers common to both. The whole structure is carried on brickwork piers, which enables the spent shale to be discharged into trucks which are run under the bench. From the top hopper, which is charged every 6 hours, the shale travels slowly down through the retort as the spent shale is discharged at the bottom by the mechanism to be described immediately, passing first through the upper zone at a temperature of about 480° C. and then through the lower zone where it is exposed to the higher temperature of about 1000° C. Thence it passes through a fire-

brick cone into a C.I. chamber, in which is situated a circular horizontal plate or table, which does not fill the chamber but which leaves an annular space. The whole column of shale in the retort is supported by the cone and by this table, and by means of the slow circular motion of an arm attached to a spindle passing up through the centre of the plate and worked by a metal bar extending the whole length of the bench and driven by motor mechanism, a portion of the spent shale, at intervals of about two minutes, is swept over the edge of the table into the annular space and so dropped into a C.I. discharge hopper from which it is emptied every four hours into trucks which run beneath it and which are conveyed by an endless chain to the spent shale bing or heap. The heating of the retort is performed by the incondensable gases produced from the shale itself in the retorting process, and in the case of the poorer shales the necessary additional gas is supplied from external ammonia-recovery gas producers of the Mond or similar type. The gases are led in a spiral direction round the retort from bottom to top in the brick chamber surrounding it, the waste gases escaping at chimneys at the top of the bench. A quantity of steam, amounting to at least 75 gallons per ton of shale, is introduced into each retort at the bottom hopper, and forms, in contact with the carbon of the hot shale at the high temperature, gases of considerable heating quality. The steam at the same time provides the hydrogen required to form ammonia from part of the nitrogen of the shale, and the excess steam and the heating gas (water gas) serve to protect the ammonia and oil vapours from decomposition by diluting them and sweeping them rapidly up out of the lower highly heated zone into the outlet pipes situated just below the upper hopper and into the large main and thence to the atmospheric condensers. These hot gases from the lower part of the retort also promote the distillation of the shale in the upper portion, and serve to equalise the temperature of the mass of shale throughout the diameter of the retort. A slight suction is maintained in the retort by means of an exhauster or fan. A single retort of this type deals with four tons of shale per day. The Crude Oil and Ammoniacal Liquor leaving the condensers flows into a separator, where the lighter crude oil floats to the surface and flows out by a pipe into a receiver, while the ammoniacal liquor, being heavier, sinks to the bottom and is drawn off by a pipe to another receiver. The uncondensed gases leaving the condensers are scrubbed with water to recover any ammonia present, and with oil to recover the naphtha which has escaped condensation. For the naphtha recovery, the gases are led up through a series of scrubbing towers filled with wooden checker-work, where they meet a descending spray of mineral oil (Shale Gas-Oil) which absorbs the naphtha. The oil is then led through a heater into a naphtha still, where a

current of steam drives off the naphtha, both being also led through the multitubular heater and thus heating the feed of oil to the stills, after which the steam and naphtha are condensed and separated, the naphtha being collected in a receiver as Crude Scrubber Naphtha ready for refining. The hot oil leaving the bottom of the still free from naphtha passes through an oil cooler and thence returns to the scrubber towers, the whole system being kept in constant circulation day and night by means of pumps. In this scrubbing plant a yield of three gallons of naphtha per ton of shale is recovered. The ammonia scrubbers are of similar structure, and the resulting ammoniacal liquor is mixed with that from the retort condensers and sent to the Sulphate of Ammonia Department for the manufacture of Sulphate of Ammonia.

The ammoniacal liquor is led into the top of an ammonia still, which is a tall, circular, iron vessel containing horizontal shelves or trays over which the liquor flows, passing from tray to tray by means of pipes. At the same time, a current of steam is passing up the still, and this, meeting the descending stream of liquor, heats the latter and liberates the ammonia as gas, which, along with the steam, passes off from the top of the still by a pipe and is led into the "Cracker." For the purpose of liberating the ammonia from the small proportion of non-volatile ammonia salts present in the ammoniacal liquor, the requisite quantity of milk of lime is pumped into the still a few plates from the top, so as to meet the liquor at a point where most of the carbonic acid has been evolved, and thus prevent the formation of carbonate of lime, which does not liberate the ammonia from the so-called "fixed" salts so rapidly as is the case when caustic lime in the form of lime-water is employed. The waste spent liquor flowing away from the bottom of the still contains the merest trace of ammonia, and the plant can be run normally for long periods absolutely free from ammonia as regards the spent liquor leaving the still. The "Cracker" is a circular wooden vessel lined with lead, and is filled with recovered acid obtained by washing with water the acid tar resulting from the treatment of the oils in the refining process. An average cracker may be charged with 2,000 gallons of recovered acid. When the ammonia and steam from the top of the still are led into this by means of a perforated lead pipe passing down to the bottom of the cracker, where they bubble up through the acid, the ammonia combines with the acid to form Sulphate of Ammonia, which remains dissolved in the hot liquid. When the acid is all saturated with sufficient ammonia, the sulphate liquor is run from the cracker into lead-lined wooden settling tanks, where any tar contained in the recovered acid is separated, and the cracker is then ready for a fresh charge of recovered acid. The product of such a charge is about $3\frac{3}{4}$ tons of sulphate of ammonia. From the settlers the clear

sulphate liquor passes into the vacuum evaporator, where it is evaporated with exhaust steam under a partial vacuum, and solid crystals of sulphate of ammonia separate out. At intervals these are dropped into a vessel below containing a perforated false bottom, where they are drained by suction and then emptied by means of a door into centrifugal machines, where they are dried, dropped into a trough provided with a spiral conveyor, and so carried through a final heated drying chamber into a hopper, whence the dry neutral sulphate of ammonia is filled into bags ready for the market. The sulphate liquor entering the vacuum evaporator, which is made of a copper alloy, must always be kept slightly acid in reaction to avoid the more active corrosion of the copper by ammonia, and in order to obtain neutral sulphate a small quantity of sulphate liquor made slightly alkaline with ammonia is poured into the centrifugal pan while in rotation, thus neutralising the excess of free acid present in the crystals, the liquor draining from the centrifugals being returned to process.

Of the Sulphuric Acid used in refining the Crude Oil, from 55 to 60 per cent. is actually recovered and used in the manufacture of sulphate of ammonia. One ton of Sulphate requires 18·5 cwts. of Acid (1·72 S.G.) for its manufacture, but in a typical Scottish refinery the recovered acid is exactly sufficient to meet all the demands for Sulphate manufacture, because that refinery is supplied not only with the crude oil and ammoniacal liquor derived from the shale retorted there, but also with crude oil from other retorting works, which do not refine the oil but which manufacture Sulphate from their ammoniacal liquor. After recovery of the acid from the acid tar, the latter still contains about 5 per cent. of acid : it is mixed with the soda tar and the mixture burned as fuel below the stills. The recovered acid from the acid tar from the refining of the Crude Oil from 5 tons of average shale will be sufficient for the manufacture of the Sulphate of Ammonia from one ton of shale.

It is the object of the Shale Oil Refiner to obtain from the Crude Oil various types of refined Oil and Wax, each possessing the physical and chemical characteristics required by market conditions, at the lowest possible working costs, and with the smallest necessary capital expenditure on refining plant, each type of refined product being produced in quantity, so far as is possible, proportional to its respective market value. The general principles that underlie the refining process, so far as the technology of the subject is concerned, are these :—(1) The separation of the complicated mixture of numerous hydrocarbons which constitute the crude oil into the fractions necessary to obtain the various refined liquid products of the standard marketable quality and value required. This separation is effected by the distillation of the oil. (2) The removal from the oil of impurities, or constituents which

must be eliminated in order to obtain products of the required quality. This purification is achieved by the treatment or washing of the oil with chemicals, and, in the case of the solid wax, by the gradual separation of oil from the crude wax by the application of gradually increasing heat, and by means of filtration through a decolorising material. (3) The separation of the crude solid wax from the liquid oils in which it is dissolved. This is accomplished by the artificial cooling of the oil to a low temperature whereby crystals of solid wax are produced which are then separated from the adherent liquid oil by filtration and hydraulic pressure.

We will now discuss the application of these principles to the actual process of refining about 65,000 gallons of crude oil daily in a typical Scottish refinery.

Distillation of the Crude Oil.

The first stage in the refining process is the distillation of the crude oil. For the separation of the various grades of oil by distillation, advantage is taken of the difference in the boiling-point of the various grades of oil, the vapour of those which boil at lower temperatures for the most part distilling over first, while those of higher boiling-point for the most part pass over later. As the specific gravity of a shale oil has a relation to its boiling-point, the separation is in practice effected by condensing the oil-vapours from the still and leading the condensed liquid oil into different receiving vessels according to its specific gravity as determined on a sample taken at the outlet of the condenser coil. The separation of the various fractions is facilitated by the use of steam, which is passed through the oil in each still. The presence of steam also serves a useful purpose in lowering the temperature at which the oil distils over, thus preventing decomposition of the oil, while it also causes a certain amount of agitation of the contents of the still and so tends to prevent local overheating of any portion. In some cases, a certain amount of decomposition is desirable, for reasons which will be stated further on, and therefore a particular stage of a certain distillation is conducted entirely without steam. The heat necessary for the distillation is supplied externally by means of a furnace under the still, except in the case of certain spirit or naphtha stills in which the distillation is effected entirely by means of steam conducted into the stills, no external heating whatever being employed. With regard to the external fuel, it is interesting to note that no coal whatever is required for this purpose, the impurities in the form of tar removed from the oil in the chemical treatment being burned under the stills, after the recovery of the available acid from the acid tar. Average Scottish Crude Shale Oil supplies sufficient tar in its refining for the supply of all the external

fuel required for all distillations of the oil, in which connection it should be remembered that, for every 100 gallons of crude oil put through the refinery, about 350 gallons of oil has to be distilled, on account of various large fractions of the original crude oil having to be redistilled. The total tar obtained in the chemical treatment of the oil and used as fuel at the stills amounts to about 19 per cent. of the crude oil (by volume), and of this about 28 per cent., or 5·36 gallons per 100 gallons of crude oil distilled, is used at the crude stills during the first distillation, the remaining 72 per cent. of the total tar being used at the other stills during the further stages of distillation.

While the purpose of distillation is to separate the different grades of oil, a certain amount of purification is also thereby effected, especially in the first distillation, where, in its later stages particularly, heavy constituents are decomposed with the separation of carbon, or carbonaceous material, in the form of coke, which is a by-product of considerable value, the resulting distillate being of lower specific gravity and of purer quality than would otherwise be the case: the quantity of chemicals required for purification is thus somewhat reduced, and a larger yield of refined products is obtained. This decomposition is also accompanied by the formation of gases, which are recovered, yielding naphtha and gas of high heating value.

For distillation, the stills used in Scottish practice are, so far as form is concerned, of two main types, known respectively as the "Boiler" and "Pot" type of still. These may be used intermittently or continuously, and separately or in combination, but for the most part the distillations are carried out in the boiler type of still. As its name implies, the boiler still is a horizontal cylindrical steel boiler provided with pipes for the inlet of the oil and for the outlet of the vapour, and with a perforated steam pipe running along the bottom of the still. The outlet vapour pipe, in which there is a hole and metal plug to act as a safety valve, is connected with a condenser, which consists of a coil of pipe immersed in a tank through which water is circulated, the vapours in the pipe (which is termed a "worm") being thus condensed into liquid oil forming what is known as the "distillate," which flows from the exit end of the pipe that projects through the side of the water tank or condenser box. The distillate drops thence through a light movable metal trough contrivance into one or other of several pipes which lead to receivers, the trough being shifted from one pipe to the other as required. Alternatively, the distillate may run from the worm-end into a flat circular C.I. pan divided into sections horizontally, each section leading into a separate receiver. This type of still is used intermittently—*i.e.*, filled up once and distilled off, for the distillation of the crude scrubber naphtha and heavy

naphtha, and for concentrating, not to dryness, certain liquid residues. A certain amount of still-liquid residue is always left in the still, as it has always been the practice in Scotland never to carry the distillation in a boiler still to the end with the production of coke, even though in small quantity, on account of the danger of thus overheating the still, though in the United States of America and elsewhere it is now, I believe, the custom in certain petroleum refineries to distil to dryness even crude oils which yield a large deposit of coke in such a type of still. In Scottish practice the residue is run off by a pipe from the bottom of the still into a receiver, either to be redistilled in the other form of still called the pot still, or to be returned and mixed with the oil at some other stage of the process.

The "Pot" type of still, which is always used where the distillation is to be carried to dryness with the formation of coke, consists of a C.I. bottom with a flange on to which is bolted a flanged cylindrical M.I. top (jointed with a mixture of fireclay, brickdust, iron-rust and salt) through which passes a steam pipe reaching nearly to the bottom of the still, and a feed-pipe for the admission of the oil. Near the top of the upper part of the still, in the side, is a door, jointed with fireclay and water, for the removal of the coke, which is removed by means of pick and shovel. From the still-head there passes the neck, which is connected with the condenser. Pot stills, or "coking" stills, as they are also called, are used intermittently for the distillation of certain residues, and in many cases, until recently, for the final distillation of the Lubricating Oil. They are chiefly used, however, in combination with boiler stills, where these are worked continuously in the first and second distillation.

The first distillation of the crude oil is carried out in such a combination of boiler and pot stills, a battery of which may consist of five boiler and twenty-eight pot stills. The crude oil flows by gravitation from the storage tanks, which are provided with closed steam coils, and situated at a higher level than the stills, into the central or middle boiler (called the feeding still), from one end of the bottom of which the residue is continuously drawn off and delivered into the opposite end of the bottom of the next of the series of boilers on each side of the central one. From each of these two boilers it is also continuously drawn off and delivered into the third of the series of boilers on each side of the central one, from each of which it is finally led off into a pipe which serves for the feeding of the entire range of pot stills, in the manner to be explained immediately. The boilers are stepped, or set in brickwork at different levels in such a way that the oil can flow from the one to the other, the flow being regulated by means of C.I. cocks. The five boilers are set in the centre of the battery, with fourteen pot

stills on either side. All the stills are heated by means of a tar spray injected and atomised with steam into the brick furnace underneath the still. From each boiler and from the pot stills a certain proportion of distillate is taken off, the heat of the furnace being so adjusted that the distillate from each still is of the desired specific gravity. By regulating the quantity of oil fed to the first still and of the residue drawn off from it and from the other stills, and by proper adjustment of the firing of the furnace and of the amount of steam introduced into the still, the specific gravity of the distillate from each still can be controlled within very close limits. The quantity of steam used varies according to the particular distillate required. If the stills are set at the proper altitudes, relatively to one another, and the conditions of flow and firing are correctly adjusted, the quantity of steam required will generally have relation to the specific gravity of the distillate—the lighter distillates may require less than 10 per cent. of steam on the distillate, and the heavier as much as 25 per cent. The condensed steam water is separated from the oil distillates by means of separators. In each still a portion of the oil is vaporised by the heat of the furnace, the vapours being carried, with the aid of the steam, by means of exit pipes on the top of each still, into the condenser, from which they pass as oil distillate through pipes into receiving vessels of about 7,000 gallons capacity.

During the distillation, the residual oil flowing off continuously from the last boiler of the series is led into one of the pot stills (previously heated and having had all air expelled and replaced with steam). The feed from the boiler is continued into the pot still for five hours, distillation taking place simultaneously. After five hours the feed is shut off that particular pot still and is delivered into another one in the series. The distillation of the oil in the first still is then continued to dryness, most of the distillate being collected along with most of the distillate from the other pot stills, and with the distillate from all the boilers except the central feeding one, the distillate from which is collected separately and constitutes the Crude Naphtha. The reason why the other distillates are all mixed after being separated is that, in the subsequent treatment with chemicals, the tar would otherwise be very thick and the temperature of the oil would have to be so high (to maintain the oil in the liquid state, on account of the concentration of the solid paraffin in the heavier distillates) that the action of the chemicals would be too energetic and so result in loss of product. The last portion of the distillate from the pot stills is collected in a separate receiver, settled well, and blown into an empty pot still, where it is carefully redistilled to dryness, the distillate being run amongst the rest of the crude distillate. If this portion of the distillate is not redistilled the crude distillate will not behave satis-

factorily in the subsequent treating or washing process, as the acid tar formed will enclose much oil, and considerable unnecessary loss of oil will result in running off the tar. At the close of the distillation in the pot stills, the then resinous-looking distillate is run down into shallow concrete wells set on the ground below each condenser worm-end. It there solidifies as a pitchy or resinous mass, which is removed to a steam-jacketted vessel, melted up and settled, and run off into barrels, or into trays to form slabs. This material is commercially known as "Shale Resin." During the distillation, the gases which are formed as the result of decomposition, especially in the coking stills, and which escape from the condenser, are collected into a wide pipe or main and are conveyed to the retorts, where they are mixed with the retort gases leaving the condensers and scrubbed for the recovery of the naphtha they contain, the scrubbed mixture of gases being then used for heating the retorts. These still gases have great heating value, consisting largely of paraffin and olefine hydrocarbons, and yielding, on combustion, from 1,200 to 1,500 B.T.U. per cubic foot. A certain quantity of naphtha condenses in the main and is collected and mixed with the rest of the naphtha for refining. The total quantity of naphtha thus recovered from the still gases amounts to 369,000 gallons yearly, or 1·82 gallon per 100 gallons of crude oil distilled. When the pot stills have been coked to dryness, they are allowed to cool and the manhole door in the side of the still top is taken off and the coke removed by a man with pick and shovel to be sent out for use in the manufacture of sodium and aluminium, the quantity of coke being about 2·5 per cent. on the crude oil. The following is a typical analysis of the Still Coke :—

	Per Cent.
Fixed Carbon	92·4
Volatile Matter	7·0
Ash	0·3
Moisture	0·3
	100·0

This coke contains about 0·2 per cent. of sulphur.

Treatment or Washing of the Distillates.

The chemical treatment of the oil, for the removal of so-called impurities, consists in stirring the oil by mechanical means or by means of compressed air for a given time with Sulphuric Acid in definite quantity in a steel vessel, and allowing it to settle. The heavy mixture of acid and impurities removed thereby, which constitutes the so-called "acid tar," is then run off. The acid-treated oil is then run into another similar vessel placed beneath

the first, and treated with solution of Caustic Soda, settled, and the so-called "soda tar" which separates out is then run off. The acid tars obtained in the treatments of the various oils at the different later stages of the refining process are mixed, and used for the first acid treatment of the once-distilled crude oil. The soda tars from the later stages are similarly mixed and used for the first soda treatment of the once-distilled crude oil. The acid used in refining is commercial sulphuric acid obtained from pyrites. It has a specific gravity of 1·84, and is as far as possible free from nitrous compounds and arsenic. The presence of selenium compounds is also injurious to the oils at certain stages. The caustic soda used in treatment is a solution of commercial caustic soda of 1·35 specific gravity. The shale oil refineries manufacture their own soda from soda ash, and some of them also produce their own acid from pyrites. "Oleum," or strong fuming sulphuric acid manufactured by the contact process, is purchased by one refinery from the manufacturers as the surplus output of the plant, when market conditions are favourable, and diluted with water at the oil refinery to the required strength. A saving on railway carriage, and by the lower price per unit equivalent of 1·84 specific gravity acid for which the oleum can sometimes be obtained, is thus effected, as compared with the purchase of the ordinary acid direct. The plant for dilution is simple and the prime cost and working cost are low. For the finishing treatment of the heavier oils, a solution of Soda Ash (carbonate of soda) replaces the solution of caustic soda. The agitation of the oils with the chemicals is effected entirely with compressed air, except in the case of the spirit, loss of which, through evaporation or volatilisation, would occur with air agitation.

The first distillation of the crude oil having been completed, the crude naphtha and crude distillate have now to be submitted to the process of purification by chemical means. The vessels in which the operation of "treatment" or "washing" is carried out are called "Washers" and are of various types. A crude naphtha washer may deal with 4,400 gallons per charge, and has a mechanical stirrer for agitation. It is a C.I. rectangular vessel with "dished" bottom, provided with inlet pipes for the oil and acid and outlet pipes for the treated oil and tar respectively. The naphtha is run into the washer, then 56 gallons (1·27 per cent. by volume) of acid is run in, and the mixture stirred for 30 minutes and then allowed to settle for an hour. The acid tar is then run off from the bottom of the washer, and the acid-treated naphtha is then run down into another similar washer placed beneath it, and 50 gallons (1·14 per cent. by volume) of caustic soda solution (1·35 specific gravity) are then run in, and the mixture stirred for 30 minutes and allowed to settle. The soda tar is then run off and

the treated naphtha is transferred by means of a pump to a storage tank for redistillation, in a boiler still, the distillate being fractionated into naphtha and light burning oil.

The type of washer used for the treatment of the Crude Distillate is a vertical cylindrical tank with a conical bottom, and is 27 feet diameter and 11 feet 6 inches high (conical part 3 feet), made of M.S. plate, and surrounded with 4½ inch brick for insulation. It can deal with 37,000 gallons of oil at one operation. There is a 6-inch inlet pipe for the oil and an 8-inch outlet pipe at the bottom, and it has also a 3-inch outlet pipe at the very bottom for the tar. A 3-inch air pipe passes from the top nearly to the bottom of the cone for stirring, and a 2-inch acid pipe passes through the side of the tank near the top and projects a little beyond the centre of the tank, to ensure the proper distribution of the acid, by means of perforations in the pipe, so that the acid is distributed over the surface of the oil and is well mixed with the whole contents of the washer by the air passing up through the oil from the bottom of the air pipe. The bottom or "soda washer" is similar, but only 10 feet 3 inches in height. There are two sets of such washers for the crude distillate, the whole being capable of dealing with 74,000 gallons of oil daily. In the washing process, the oil, at a temperature of about 50° C., is run from the elevated charging tank, into which it has been pumped from the crude stills receivers and in which it has been kept warm and has been settled by means of closed steam coils, into the acid washer, where it is agitated with air while about 900 gallons (2·38 per cent. by volume) of the acid tar derived from the subsequent stages of refining is run in, along with 0·75 per cent. of acid. The stirring is continued for 40 minutes, and, after settling for 15 minutes, the acid tar is run off into a tar box, which is a wooden vessel lined with lead, from which it is run down into an acid tar blower, which is a horizontal boiler with air pipe, from which, by means of compressed air, the tar can be blown to a series of Tar Boxes for the recovery of the acid—a process which will be presently described. Another portion of acid (0·63 per cent.) is then run into the washer and stirred for 40 minutes. It is allowed to settle for 7 hours, and the tar is then run off into the tar box as before. The acid-treated oil is then run down into the soda-washer, where all the soda tar available from the later stages of refining is added, and the mixture stirred for 40 minutes. In some cases a little caustic soda solution is also added from an elevated Soda Box. The oil is then settled over-night and the soda tar is run off into a soda tar blower. The treated crude distillate (or "Green" Oil as it is called, from its more or less green colour) is then pumped away to storage tanks, ready for the Second Distillation. The loss in volume of the crude distillate incurred through the removal of impurities as tar in the washing

process at this stage amounts to about 13·5 per cent. on the acid treatment and about 4 per cent. on the soda treatment.

The stills employed for the second distillation, or the distillation of the treated crude distillate, are similar in design and arrangement to the crude distillation plant, the treated crude distillate being run by gravitation from the storage tanks into the first of a series of boiler and pot stills, in which latter the distillation is finally carried to dryness with the formation of coke. A more recent mode of operation, however, is to draw off continuously the residue from the last boiler still and return it to the crude oil, so that the use of pot stills in the second distillation is avoided, thus effecting a considerable saving, as the frequent renewal of the still bottoms is an important item in the refining costs, the average life of a still bottom being one year. This second distillation is conducted somewhat differently from the first or crude oil distillation. Owing to the purification already effected by the first distillation and treatment, the pot stills can be fed continuously for 7 or 8 hours, instead of 5. In order to obtain a more crystalline solid paraffin which may be easily extracted by the process of filtration to be afterwards described, the later stages of distillation in the "Green" pot stills are conducted entirely without steam. The fractions obtained at this stage are :—

(1) Crude Burning Oil.
(2) Heavy Oil and Paraffin.
(3) Residuum, returned to the Crude Oil.

The Crude Burning Oil is now treated with 0·67 per cent. of acid and with 0·75 per cent. of caustic soda solution, stirring 30 minutes in each case, after which the treated oil is redistilled in a series of boiler stills, termed Fine Oil Boilers, the residue from the last boiler being run off continuously through a cooler into a receiver. The fractions obtained here are :—

(1) Lamp Oil or Power Oil.
(2) Lighthouse Oil.
(3) Residuum.

The Residuum is blown into a single boiler still and redistilled, giving heavy burning oil, light gas oil and heavy gas oil. The lamp oil or power oil and the lighthouse oil are blown to storage tanks ready for the market.

The Heavy Oil and Paraffin portion of the distillate is more or less solid at ordinary temperatures, owing to the presence of about 25 per cent. of solid paraffin, and the next stage in the manufacturing process is the separation of this solid paraffin.

Extraction of the Solid Paraffin.

This is accomplished by cooling the heavy oil and paraffin artificially to cause the formation of crystals, and subsequent filtration to separate the crystals from the liquid oil. The cooling is first conducted in open pans under a roof without sides, or by passage down a tower through which a current of air created by a fan is passing upwards, and, being thus cooled to about the temperature of the air, it is then pumped through the coolers, which are the refrigerating vessels for the cooling of the oil to very low temperatures by artificial means. A single cooler, of a type frequently employed, consists of a C.I. tubular casing about 20 feet long and 20½ inches in diameter, placed horizontally, and containing within it a steel tube 15 inches in diameter, the annular space between the inner tube and the outer casing or jacket forming the space for the circulation of the liquid ammonia from the Compressor. Through the centre of the inner tube from end to end there passes a steel shaft of 4 inches diameter on which are fixed scrapers which serve to scrape off the frozen oil from the inner sides of the cooler, and so renew the coating. The ammonia enters by a half-inch M.I. pipe, and the vapour leaves by a 3½-inch outlet. The oil enters and leaves the cooler by 3-inch pipes. Into the outer jacket of the cooler liquid ammonia is pumped under pressure from a compression engine. This compressor, charged with anhydrous ammonia liquefied under pressure and received into the works in steel cylinders, pumps the liquid ammonia into the jacket of the cooler, where the ammonia passes from the liquid into the gaseous state owing to the diminution of pressure (ammonia being a gas under atmospheric pressure) heat being necessarily absorbed in the process, and as the only source of heat available (Heat and cold being merely relative terms) is the oil in the inner body of the cooler, the result is that heat is transferred from the oil to the ammonia and the oil is thereby cooled. In this way it is possible to cool the oil to a temperature approaching $-20°$ C. The actual temperature employed in the ordinary practice in shale oil refineries ranges from about $0°$ C. to $-10°$ C., the temperature being regulated as desired by the amount of oil pumped through the cooler, or, what is equivalent, by the time during which the oil is in contact with the cooler. The gaseous ammonia leaving the jacket of the cooler returns to the compression engine, where it is liquefied, the liquid ammonia being then led through a series of cooling pipes or condensers, cooled by water, in order to absorb the heat evolved on the compression of the ammonia in the passage from the vapour to the liquid phase, after which the cooled liquefied ammonia passes into the cooler jacket where the oil is cooled and the ammonia partially vaporised, the vapours being

returned to the compressor, and the cycle of processes thus going on continuously. There is a certain unavoidable loss of ammonia due to leakage, so that while, theoretically, when once the coolers are charged with liquid ammonia, the cooling operations should go on for an indefinite period without the addition of fresh liquid ammonia, as a matter of fact, during a single year, in one refinery, the quantity of anhydrous ammonia used was equal to 36 pounds per million gallons of crude oil put through the refinery.

The coolers in one refinery are arranged in sets of four, and one such complete cooler, containing four sections, is capable of cooling 11,000 gallons of heavy oil and paraffin from 20° C. to 0° C. in 24 hours, and four such coolers can deal with the daily throughput of heavy oil and paraffin, which at this stage is cooled to about 0° C. The temperature of the oil leaving the cooler and of the oil leaving the filter presses are guides to the efficient extraction of the quantity of solid paraffin desired and to the resulting "setting point" of the filtered oil, the setting point being briefly described as the lowest temperature to which the oil can be cooled without visible separation of crystals of solid paraffin causing a cloudiness in the oil. The heavy oil and paraffin having been cooled to the required temperature, the next stage is the separation of the solid paraffin from the liquid oil. Under the pressure of the same pump which pumps the oil through the cooler, the cooled oil leaving the cooler is forced through a pipe into a series of Filter Presses. A filter press may be of any convenient length, depending upon the number of filter plates of which it is built up. The presses used may contain from 50 to 180 plates each. For the throughput we are considering, 42,000 gallons of heavy oil and paraffin, 1,403 filter plates are employed. These are set vertically in a C.I. framework erected sufficiently above ground level to allow room for a shallow metal trough fitted with a spiral conveyor running along under the bottom of the filter press, so that when the filters are emptied the cakes of solid paraffin will fall into the trough and be conveyed by the action of the conveyor to the floor of the hydraulic press department. The plates are hung by snugs on each upper side to the upper sides of the framework, which is somewhat longer than the combined length of all the plates when in position, so that for the purposes of emptying, each plate can be moved along the framework sufficiently apart from its neighbours to allow of the cake of solid paraffin being dropped or scraped off into the trough beneath. The plates are corrugated, with a spout or outlet at one of the lowest corners for the outflow of the filtered oil into another narrow trough which runs along the length of the filter press just below these spouts. Each plate has a hole right through its centre, by means of which, when the plates are close together, the oil from the cooler can pass from one to the other, filling up the spaces left

between the recessed or concave or hollowed outer sides of each plate. Over the top of each plate there is folded a sheet of strong filter-cloth, and the plates are screwed up close together by a powerful hand screw or by hydraulic pressure applied at one end of the filter press. Under the pressure of the pump, amounting to a maximum of 100 lbs. per square inch, the cooled oil is forced through the filter cloth and passes out through the hole at the bottom corner of the plate into the spout, whence it flows into the trough leading to a receiver. The mass of crystals forming the solid paraffin, on the other hand, is retained between the surfaces of the cloth covering adjoining plates, and after six hours, when the filter presses are ready, the feed of oil from the cooler is shut off, and the plates, which have till now been kept close together by the powerful screw, are slid along in rotation by the men (the screw having been slackened) who loosen the cakes of solid paraffin (in an oily condition) from the cloth with blunt knives, thus dropping them into the trough below. The plates are then slid back close together and pressed tight by the screw so as to keep the surfaces of the cloth-covered plates so close together as to resist the escape of any of the oil and paraffin through any but the proper channels. The feed of oil from the cooler is then again allowed to enter the presses, while the other half of the presses are being emptied. The filter-pressed cakes of paraffin still retain much oil, and for its further removal the mass of paraffin is packed in strong twilled hempen or cotton cloth and submitted to a much higher pressure in hydraulic presses. The packing of these presses is carried out thus: one of the latticed plates made of plaited strips of thin steel forming a sort of close lattice-work and measuring 3 feet 4 inches by 2 feet 6¼ inches, is laid on the floor. A square of strong specially-woven cloth is laid upon it, a wooden frame is laid on it, and the mass of soft paraffin is shovelled on to the cloth inside the frame and smoothed flat, forming a layer about 2 inches thick. The purpose of the frame is to prevent spreading of the material to the outer edges of the cloth. The cloth is now folded over at the sides and ends, and on this another lattice work tray is laid. This is also covered with a cloth, the wooden frame is laid upon it, and another layer of soft paraffin shovelled on to it as before, and these operations are repeated till the pile of trays belonging to the press is complete. They are then lifted up, one by one, and each is placed on one of the shelves of the hydraulic press. These shelves consist of strong steel plates, which rest on supports at each side of the framework, the supports being designed so that, when hydraulic power is applied to the ram at the bottom of the press, the iron shelves carrying the latticed plates of soft paraffin slide in a level horizontal position up the supports, and being met with the resistance of the strong fixed plate forming the top of the framework of the press

the resulting pressure expels the greater proportion of the oil, which oozes out through the cloth and drips over the sides of the shelves into a well sunk in the floor, whence it is returned to the heavy oil and paarffin storage tanks. In the packing operations the men work together in pairs. As soon as the packing of the press is completed, the hydraulic pump is started and pressure is gradually applied till the maximum pressure of 1 ton per square inch is attained. The presses then remain under that pressure for six hours in all, when the pressure is released, the plates descend by their own weight, the latticed plates are removed by the men, the cloths are unfolded, and the cakes of solid paraffin are lifted on to a flat barrow and wheeled away and emptied into a large Melter, which is a horizontal boiler with a large hopper, situated below ground level and provided with open steam pipes, where it is all melted up and pumped away to a storage tank ready for refining. This crude solid paraffin wax is technically known as "Scale" from its crystalline, flakey appearance. It has a melting-point of about 46° C., and contains from 5 to 10 per cent. of oil. The empty hydraulic presses are packed with the plates which have been previously charged with a fresh quantity of the filter-pressed paraffin. The oil which drains away from the hydraulic presses runs off from the wells situated beneath the presses into receivers, from which it is pumped back to the heavy oil and paraffin storage tanks, because, the temperature having risen considerably in the hydraulic presses, the oil still contains more solid paraffin than it ought to do. The oil running from the filter presses, on the other hand—termed "Blue Oil," from its colour—is collected in a receiver and pumped to a storage tank, ready for treatment. Sixteen hydraulic presses are used for the heavy oil and paraffin, each having 25 plates, thus pressing 400 cakes in all every six hours, or 1,600 cakes per 24 hours. Each cake weighs about 32 lbs., so that the total crude solid hard paraffin turned out from these presses is about 23 tons daily.

The Blue Oil is treated with 0·45 per cent. of acid and with 0·4 per cent. of caustic soda solution, being stirred with each for 30 minutes. The well-settled treated oil is then transferred to a storage tank provided with closed steam coils to ensure thorough settling, ready for distillation in the Blue Oil Stills. It may be distilled in various ways. The older method, still in use, is that of first distilling off (1) Heavy Burning Oil, (2) Light Gas Oil, and (3) Heavy Gas Oil in Blue Oil Boiler Stills worked either continuously or intermittently, the Residue from the last boiler still being run off (continuously or intermittently) through a water-cooled coil of piping into a storage tank, from which a number of pot stills are charged daily, each being distilled down to dryness with the formation of coke, separating the distillate into :—

(1) Heavy Gas Oil;
(2) Cleaning Oil;
(3) Lubricating Oil (Light) and (Heavy); and finally
(4) A so-called Residuum Oil.

In some cases a certain proportion of caustic soda liquor is run into the pot stills during the distillation of the lubricating oil fraction for the purpose of improving the colour of the oil, but this is certainly not an infallible means of obtaining pale-coloured finished Lubricating Oil.

During the distillation of the blue oil, great attention must be paid to the regular firing of the stills and to the proportion of steam employed, in order to secure distillates of the viscosity, flash point and colour required. To obtain the high flash point for a heavy lubricating oil (180° to 190° C.) it is imperative that the distillation should proceed slowly and regularly throughout, as the production of even traces of light vapour through partial decomposition due to overheating is sufficient to appreciably lower the flash point of the distillate, and want of attention to these points leads to disastrous lowering of the flash point. By the use of an "atmospheric" condenser, or by means of a system of "pockets" in the condensers employed in one of the refineries, distillates of good colour and high flash point can be obtained with less difficulty, as the lighter vapours which would otherwise lower the flash point pass on further in the condenser and so can be collected separately from the main body of the distillate. The "pocket" system consists in leading a pipe, from a point about half-way along the condenser worm, which leads the heavier distillate through the side of the condenser tank to be collected in the receiver, while the lighter and more volatile distillate passes on as vapour and is condensed in the second half of the condenser worm and runs out at the outlet to be collected separately. The firing of the stills during the lubricating oil stage should be so regulated that the distillate at the worm end will increase only about 3 degrees in specific gravity per hour.

Where the Atmospheric Fractionating Condenser is employed, the blue oil passes through a series of boiler stills, where distillates consisting of (1) Heavy Burning Oil, (2) Heavy Gas Oil, and (3) Cleaning Oil are obtained. The vapours of the distillate from the fourth boiler still are led through the atmospheric condenser, which consists of a series of vertical inverted U-shaped pipes, arranged in sets, each set communicating with a chamber beneath, so that the less volatile heavier portion of the distillate condenses in the earlier of these chambers, and the more volatile lighter portion passes on to the succeeding chambers. In this way the vapours from the still can be separated by fractional condensation into (1) Heavy

Lubricating Oil Distillate, (2) Light Lubricating Oil Distillate, and (3) Heavy Cleaning Oil. The Residue (·920 to ·925 specific gravity) from the fourth boiler still is run off continuously as Residuum Oil, which is well settled in a receiver with closed steam coils and is then ready for the market as "Residuum Oil," largely used for grease-making and as a wire-rope oil. Alternatively, it can be redistilled in pot stills, yielding more lubricating oil and a still heavier and more valuable residuum oil (·930 to ·940 specific gravity).

The Gas Oil, Cleaning Oil and Lubricating Oil distillates from the blue oil stills still contain large quantities of solid paraffin varying from about 7 to 13 per cent. in the various fractions, most of which can be separated by further cooling and filtration. For this purpose each of these distillates is blown to separate storage tanks, from which they are passed through separate coolers, filters, and hydraulic presses, for the separation of the solid paraffin. The filter presses for all these are worked up to a pressure of 100 lbs., and the hydraulic presses to 1 ton per square inch for the Gas Oil and Cleaning Oil, and to 15 cwts. for the Lubricating Oils. They are emptied every six or eight hours. The solid paraffin from all these hydraulic presses is melted up along with that from the heavy oil and paraffin presses (with the exception of sufficient gas oil paraffin for the manufacture of special low-melting point "Miners' Wax" and "Match Wax," melting point 37° to 39° C.) and the mixture is pumped away to the Paraffin Wax Refinery for the production of Refined Wax. The gas oil, cleaning oil and lubricating oils from the filter presses are collected in separate receivers. The gas oil is blown to the Finished Gas Oil stock tank ready for the market. The Cleaning and Lubricating Oils are blown to washers holding 4,000 gallons each, where they are treated in a special way, which varies in different refineries and under different circumstances: one method for the treatment of lubricating oil may be described here. The oil is agitated with 2 per cent. of acid, and after short settling the tar is run off and the oil again agitated with 2 per cent. of acid, and the bulk of the tar is run off after short settling, and the remainder several times at intervals during the day, after which the oil is allowed to settle for 24 or 48 hours as is necessary, until it is quite free from tar, which is carefully drawn off. The oil is then dropped from the acid washer into the soda washer beneath it, where it is gently turned over by means of air agitation with a quantity of cold water which has been added in the soda washer, after which the watery layer is run off. Seventeen per cent. of soda ash solution (carbonate of soda solution of 1·02 specific gravity) is then run in and the mixture is gently stirred with air until the smell of the oil is quite sweet, and all traces of acidity have disappeared. After steaming to 52° C. the soda liquor is drawn off and the oil is washed with water, after which it

is blown to settling tanks where it usually settles perfectly clear and bright in two days, being then pumped to the storage tank as Finished Lubricating Oil ready for the market. It is most important that the lubricating oil should be treated at the proper temperature, which depends upon various considerations, but in any case the temperature should be as low as is consistent with the subsequent thorough settling perfectly free from acid tar granules without too great an interval of time intervening between the acid treatment and the neutralisation of the oil with the alkaline solution.

In the removal of the solid paraffin from the distillates the specific gravity of the oil is raised, as the solid paraffin has a lower specific gravity than the oil which accompanies it in the distillate. Gas Oil rises about 10 degrees and Lubricating Oil about 5 degrees in specific gravity. By the chemical treatment the specific gravity is lowered, as the tar impurities have a higher gravity than the purified oil. The Cleaning Oil gravity falls 2 or 3 degrees, and the Lubricating Oil 5 or 10 degrees on treatment. The flash point may be slightly lowered by removal of the solid paraffin, and slightly raised by treatment, while the viscosity of the oils is somewhat increased by extraction of the solid paraffin, and somewhat lowered (2 degrees for Light and 4 or 5 degrees for Heavy Lubricating Oil) by the chemical treatment. For all these reasons it will be apparent that in the cutting out or separation of the different distillates at the blue oil distillation, in which these are separated according to the specific gravity of the distillate as it runs from the worm end of the condenser, this increase and decrease in specific gravity (and to a lesser degree in flash point and viscosity) must be allowed for, in order to ensure that the finished product will be up to standard quality in these respects.

Refining of the Crude Solid Paraffin.

The first stage in the purification of the solid paraffin is the process called "Sweating," and is carried out in so-called "Sweating houses." The storage tanks containing the melted crude paraffin, and the "Strainings" (or "sweatings" from previous operations), are placed at an elevation above the sweating houses, which are built of brick and may measure 52 feet by 16 feet by 12 feet high, and may consist of two storeys. The house is furnished with closely fitting iron doors at each end, and on each side of the house, along the inner wall lengthwise, there run a series of steam coils by means of which the temperature of the inside of the house can be regulated. Supported by racks made of iron piping, there are erected a number of large shallow iron trays, about 21 feet by

6 feet 6 inches area and 6 inches deep, placed horizontally one above the other, and resting on transverse steam heating pipes fitted in vertical standards. By means of an ingenious arrangement of lever and eccentric, the trays or pans are so balanced that they can be slightly lowered at one end or raised again to the horizontal position. In each tray, two inches from the bottom, rests a false bottom consisting of lattice-work made of thin strips of metal plaited together. With the trays in a horizontal position, and by means of the short branches of a vertical pipe fitted with stopcocks running up one end of the trays in a central position, water from a pond is admitted so as merely to reach the false bottoms of the trays, and the melted crude solid paraffin, mixed with sufficient sweatings to reduce the melting point of the mixture to about 42° C., is run in from the charging tanks by means of another pipe on to the top of the water until the trays are full. The doors of the house are left open until the melted material has cooled and become solidified, after which the trays are inclined by means of the lever and the water is run off by the outlet pipe placed at one end of the trays into iron cups communicating with a vertical pipe connected with a horizontal pipe which runs off to the pond and to receiving vessels as required. The water is run off to the pond to be used again, and the cakes of crude paraffin are thus left resting on the false bottoms of the trays. The doors of the sweating house are then closed, and the temperature is gradually raised by means of the closed steam coils. As the paraffin is heated it expands and becomes porous, and the yellow oil which it still contains, along with the portions of paraffin of lower melting point, gradually oozes out, finds its way into the space below the false bottoms of the trays, and runs away by the short off-take pipes into a receiving vessel. The solid paraffin, through the removal of the oil and some of the paraffin of lower melting point dissolved by it, thus becomes whiter in colour and of higher melting point. The oil which first leaves the sweating trays is collected in the receiver (up to 32° C., setting point) and re-sweated by itself in a separate sweating house, yielding "Residue Oil" which is returned to the oil refinery, and soft wax of very low melting point. The next fraction oozing from the trays is called "Strainings" or "Sweatings," and is partly returned to the crude paraffin to reduce its melting point, and partly re-sweated yielding more sweatings and soft wax. When these have been collected, and when the colour and melting point of the sweated paraffin left in the trays are as desired (the melting point being about 47° C.) the temperature of the house is quickly raised and open steam admitted by the branches of the steam pipe (which is used in common for steam and water) until the whole of the wax is melted and run off by the pipe into a separate receiver. This is known as "Once-sweat Wax." The

reason for reducing the melting point of the crude paraffin by admixture with sweatings to 42° C. is to leave a sufficient range of melting point between the material as charged into the sweating house and the once-sweat wax to allow as much as possible of the colouring matter and oil to be removed without undue loss in yield of sweated wax through the removal of too much solid paraffin with an accompanying too great rise in its melting point. For the same reason, the sweating process must not be rushed too rapidly, but time ought be given to allow for the gradual elimination of the yellow oil so as to get the maximum yield of wax of the best possible colour left in the trays.

The once-sweat wax, after separation from the water in the receiver, is pumped up into the trays of another sweating house, to be again cooled and re-sweated, yielding strainings of soft wax and leaving in the trays hard wax of good colour and of melting point either 48° or 50° C. (two common marketable grades) or indeed any special melting point required up to 55 C°., though in this case the yield of finished wax is very small. The hard wax is melted up and run out and collected in another receiver. On account of the long time required (especially in warm weather) for the cooling of the crude paraffin in the trays, a large number of such sweating houses are required, twenty-four being employed for the throughput here dealt with.

Each grade of wax as it leaves the sweating houses is steamed with open steam in the receivers and thoroughly settled, after which it has to be further refined by filtration through a decolorising material. For many years a very good agent for decolorisation was found in the so-called "Prussiate Char," a by-product from the manufacture of yellow prussiate or ferrocyanide of potash. In this case the melted wax was agitated by mechanical means with one or two per cent. of the prussiate charcoal. More recently the mediums used have been such materials as "Eponite," made from a mixture of fuller's earth and a highly active wood charcoal, and "Norite," which is much used at the present time in sugar refining, though it may here be pointed out that a material which may be excellent as a decoloriser for other substances may be very inefficient as a decolorising agent for wax. Such materials as the above were stirred with the wax by mechanical means or by agitation with air, and it was most essential that they should be thoroughly dried and in a fine state of division, but the best of them frequently failed to give a pure white wax in practice, whatever the proportion used, a greenish tint being always observable when the large cakes of wax were piled up in bulk.

Great success, however, during the last ten years has attended the use of a material called "Floridin." This is a particular form of fuller's earth found in Florida, U.S.A. It has the remarkable

property of absorbing and thus removing the colouring matter from oils and wax which are filtered through it. It is used in a granulated form (16 to 30 mesh per inch) and is previously ignited at a temperature of about 500° C., it being most important that all moisture, free or combined, should be driven off before use. While, in a finely powdered condition, Floridin removes much of the colouring matter of the wax when stirred with it, by far the best results are obtained by filtration through a long column of the material. The Filter employed consists of an upright cylinder, made of M.S. plate 7-16ths inch thick, 14 feet 3 inches in height and 2 feet 6 inches in diameter, the lower half being jacketted and having half-inch steam inlet and outlet pipes to keep the wax in a melted condition and at a comparatively high temperature during the filtering process. There is a 2-inch inlet pipe for the wax at the top and a similar outlet pipe for the filtered wax at the bottom. On the bottom of the cylinder there is a sloped casting with a vertical door 2 feet 5 inches long and 9 inches high.

Level with the bottom of the door there is a perforated M.S. plate on which a cloth is laid to enable the filtered wax to run through while retaining the particles of Floridin. The previously ignited Floridin is run from a hopper into the filter, which holds about a ton. The sweated wax is then run in from a storage vessel placed above the filter, and allowed to percolate slowly down through the mass of Floridin. The rate of flow must be adjusted so as not to be too rapid, as time must be allowed for the decolorising effect to be produced, and the presence of moisture in the melted wax and in the Floridin must be rigidly guarded against. The upper surface of the Floridin in the filter must also always be kept covered with the entering wax during the process of filtration, in order to exclude the presence of moist air. Samples are taken from the wax outlet pipe as it delivers through a cloth filter screen into a receiving vessel, and when the colour begins to show a yellowish tint or when the bulk of the filtered wax is of the correct standard colour the flow of wax entering the filter is cut off. The white filtered wax is then pumped to a storage tank and run into trays arranged in racks, from which the cakes of finished wax are turned out next morning and packed in bags or wooden cases ready for the market. It is found best first to run the hard wax through the filter, and then to run the soft wax through, as the Floridin, after its decolorising power for securing the standard colour of the hard wax has been exhausted, is capable of decolorising the soft wax up to the lower colour standard required for the latter.

When the filtration of the wax has been completed up to standard colour, the filter is allowed to drain into a separate receiver, the process being accelerated by air pressure applied to the top of the filter (which is supplied with a top cover for this purpose), after

which the bottom door is opened and the spent Floridin allowed to run out. It consists of about 70 per cent. of Floridin and about 30 per cent. of wax and colouring matter. It is removed in barrows or by a conveyor to the Extractor, where the adhering wax is recovered by extraction with hot naphtha. The extractor itself is a vertical cylindrical steam jacketted vessel divided horizontally into two divisions by a steel piate, these divisions being connected by a syphon pipe. The upper portion is charged with about 2½ tons of the exhausted Floridin from the filters (of which there may be four) through a manhole door on the top, and the door is then closed down. A sufficient quantity of naphtha is then run into the extractor from a naphtha charger situated above, by means of a pipe. Through the agency of a closed steam coil in the lower division the naphtha is vaporised and the vapours pass up by a passage at the back of the extractor and are there condensed at the top of the upper portion by a pipe through which cold water is kept circulating. The hot condensed naphtha drops down and percolates through the mass of Floridin, flowing off by means of the syphon pipe into the bottom division, where the naphtha is again vaporised and again passes up as vapour to the top of the extractor, where it is again condensed and percolates down through the Floridin to be again syphoned off, and this circulation goes on repeatedly and automatically until a test of the naphtha from the syphon proves that no more wax is being dissolved, and that the Floridin has therefore been thoroughly extracted. Open steam is then admitted to the bottom division and to the upper division until all the naphtha has been vaporised and driven off both from the wax and from the Floridin ; in this case the naphtha vapours being swept right out of the extractor are condensed in an external condenser, collected in the naphtha storage vessel, and are ready for use for a fresh charge of Floridin. The extracted wax and colouring matter, dissolved by the hot naphtha, and being much less volatile than the latter, are left behind in the bottom division of the extractor, whence they are run off into a receiving vessel, from which they are pumped back to the sweating house to be re-sweated. While the naphtha (a special fraction which contains the minimum of very volatile constituents to avoid loss through insufficient condensation, and the minimum of high boiling constituents which might impede the rapid vaporisation of the naphtha in its repeated cycles of vaporisation and condensation during the extraction process) is an excellent solvent for the wax, and while it also removes a considerable proportion of the colouring matter, some of the latter always remains, and to remove this effectively in order to restore the Floridin, as finally recovered, to its original effectiveness, various other organic solvents have been tried experimentally during the last ten years. Amongst these may be

mentioned carbon tetrachloride, alcohol, and caustic soda solution. All of these have been found to dissolve more or less of the colouring matter, which is insoluble in hot naphtha, but only the use of alcohol in some form has been found to restore the original efficiency of the Floridin, though its application has not yet been adopted on the manufacturing scale.

The extracted Floridin from the upper division of the extractor, containing not more than 0·3 per cent. of wax and colouring matter, is removed by a man-hole door in the side of the extractor on to the floor, whence it is conveyed in barrows, or may be conveyed by an elevator, to the Rotary Furnace. This is an inclined horizontal cylindrical steel tube (like a Lancashire Boiler flue) lined with firebrick, 25 feet long and 2 feet 6 inches in diameter, which is made to revolve by a motor-driven mechanism at a speed of 8 revolutions per minute. The Floridin from the extractor is introduced into the upper end of the furnace and travels down to the lower end, whence it is discharged by means of an opening into an inclined shoot or trough from which it delivers into a pipe, through which, by the suction of a Sirocco Fan, it is drawn up into the charging hoppers above the filters ready for charging the filters again. The heating of the furnace is attained by means of a brick oven in which a mixture of still coke and smithy coke is burned, the hot gases therefrom passing through the rotary furnace and thus supplying sufficient heat for the ignition of the extracted Floridin, so that any traces of naphtha, wax or colouring matter, as well as all moisture, are driven off or decomposed, being ignited at a temperature of about 500° C.—a temperature wh ich might be advantageously reduced considerably provided the time of heating were extended, or the perfect combustion of the carbonaceous residue completely secured. The recovered Floridin from the furnace has somewhat less than half its original decolorising power, but can be used continuously for years on end without further deterioration, except through slight mechanical loss and production of dust which has to be rejected. From our experience of the use of Floridin, extending over ten years, we know that an original charge of Floridin can be repeatedly recovered, and that such recovered Floridin can be repeatedly used throughout very long periods, for during an entire year we actually used only recovered Floridin (except 7 tons of fresh material to replace mechanical loss) for the production of 7,341 tons of Finished Refined Wax of the same excellent quality and standard colour obtained when using only fresh Floridin. The deciding factor in the question as to whether it is more profitable to use only fresh Floridin or only recovered Floridin or a mixture of both depends on the capacity of the filtering and related plant available, and on the relative values or prices of naphtha and Floridin respectively. With fresh

Floridin a smaller percentage is required, and therefore less naphtha is required for its recovery; so if naphtha be cheap it pays best to use only recovered Floridin repeatedly, while if naphtha be expensive then it is more profitable to use fresh Floridin, or at least a proportion of it only, at regular intervals, say weekly or monthly. It has been found that, with Floridin ranging from £2 to £20 per ton, with naphtha at 1s. 7d. per gallon, or with naphtha ranging from 6d. to 3s. per gallon, with Floridin at £13 per ton, the total cost of decolorising with recovered Floridin is always less than when using original Floridin only.

A mineral named Bauxite, well known as a source of aluminium and its compounds, has been employed to a considerable extent for decolorising wax derived from petroleum, as well as for the partial removal of the sulphur from certain liquid petroleum products, but this material, in our experience, possesses certain disadvantages which do not attach to the use of Floridin, although it is less costly, ton for ton, than the latter.

As this paper has been designed to deal with the technology of the manufacturing processes, the characteristics and uses of the various marketable products have not been dealt with, but it may be stated that the yields of these products depend both on the character of the shale and on the treatment it receives in the retorting process. In actual industrial practice, the yield of crude oil, including naphtha, may vary, in two areas working different shales, from 18 gallons to 26 gallons per ton; while the yield of ammonia, from the same two areas, may vary from 51 lbs. to 23 lbs. per ton, expressed as commercial sulphate of ammonia. The average all over the industry may be taken at about 20 gallons of oil and 40 lbs. of sulphate of ammonia per ton of shale retorted.

With regard to the yields of refined products obtainable from the crude oil itself the following comparison may be of interest:—

COMPARISON OF YIELDS OF REFINED PRODUCTS FROM CRUDE OILS FROM SHALES FROM DIFFERENT AREAS.

Particulars.	Area A.	Area B.
Sp. Gr. of Crude Oil	·891	·865
Setting Point of Crude Oil	87°F.	85°F.
Refined Products.	**Per Cent.**	**Per Cent.**
Naphtha	0·96	0·60
Burning Oil	22·85	26·00
Gas Oil	13·79	16·67
Cleaning Oil	7·05	7·75
Lubricating Oil	11·34	9·94
Refined Wax	9·41	15·09
Residuum	1·32	1·58
Total Refined Products	66·72	77·63

These instances are extreme ones, but they may serve to illustrate the striking differences in the yields of the various products, and in the total refined products, when retorted in the same retort, employing shales of different character from different mining areas. The differences resulting from the effect of varying temperatures upon the shale in the course of the retorting process are equally striking and fundamental, when carried out on the same shale, and this and similar other aspects of retorting may be dealt with at some future time.

Note on the Effect of Small Quantities of Volatile Impurities on the Flash Point of Kerosene.

By E. C. Craven and B. G. Banks.

In connection with Colloidal Fuels, it has been suggested (*) that the addition of finely divided coal, with other carbonaceous material, to an oil, raises its flash point. In carrying out some work on such mixtures we found that the flash point is in some cases raised. This rise may be due to several causes, as for example:—

(a) Presence of moisture in the added solid material.
(b) Decreased proportion of volatile constituents.
(c) Selective absorption of volatile constituents by the solid material added.

With reference to the last cause, it has been suggested by Dr. Ormandy that, if this were true, a finely divided inert solid material, such as sand, clay, or the like, would have a similar effect to carbonaceous substances.

Experimental. In the present work most of the flash point determinations were made by means of the Abel Tester. The thermometer was checked against standard instruments, and found to agree to 0·2° F. After various trials it was found that the best method of arriving at the true flash point of mixtures was as follows :—

A preliminary test was made, in which the test flame was applied in the ordinary manner, once for each degree rise in temperature until the flash point was obtained. The second test was then made, the test flame not being applied until the temperature had

* *Cf.* L. W. Bates. U.S. Patent No. 31634/1920.

risen to within 5° F. of the flash point temperature previously found. In general, the second test gave results 1° F. lower than the first test. This is, without doubt, due to the loss of vapour which occurs on repeated opening of the test flame slide.

The effect of the rate of heating was also examined, and it was found that consistent results were obtained when the temperature of the oil cup rose 3° per minute. It is sometimes recommended that for oils having a flash point higher than 120° F. water should be used between the test cup and the water bath, but no advantage or difference in results was found owing to such a procedure, so long as the rate of temperature rise previously mentioned was adhered to. The oil or mixture was stirred steadily throughout all determinations.

Repeat tests were conducted in practically all cases and mean figures are given. Repeat tests commonly agreed to 1° F., except in mixtures to which much gas had been added.

In shaking samples with clay or the like the clear liquor only, after settling out the solids, was used for the flash point determinations. A blank test was done, where necessary, to determine the rise in flash consequent on the shaking in a partly filled bottle without clay, and a correction made where such rise was found.

The coal used was Derby house coal, ground and dried at 80° C., and passing a 160 mesh sieve. The clay used was a fairly fine china clay dried at 105-110° C., and passed through a 60-mesh sieve.

	Flash point.
"White Rose" Kerosene (A)	120° F.
" Ormandy and Craven test*	120° F.
Mixture of 60% wt. ditto + 40% wt. coal	122° F.
" 60% wt. ditto + 40% wt. clay	122° F.
Kerosene after shaking with 40% clay	121° F.

A slight rise in flash point thus seemed to be shown. In order to test the hypothesis that this rise might be due to decreased proportion of volatile matter in the test cup, experiments were made in which some of the oil in the cup was replaced by pieces of glass rod. The results were as follows :—

		Flash point.
52% kerosene ..	48% glass	121° F.
37% kerosene ..	63% glass	120° F.

Practically no effect thus seems to be produced by a diminution in volume of the kerosene exceeding that occurring with the clay

* The flash point as determined by the glass instrument described *J.* VIII. 31, p. 151. The characteristic difference of this instrument is that the volume relation liquid-air is much lower than in the Abel.

mixture. As dry materials were used, the moisture hypothesis is untenable, so that we are forced to consider the selective absorption explanation. It will be noticed that a slight rise in flash point was obtained when the kerosene was merely shaken in a closed bottle, leaving only a small air space, with 40 per cent. of its weight of clay, and the clear oil tested after the clay had settled out.

By good fortune a second sample of the same brand of kerosene was now obtained, which showed these differences in a greater degree.

	Flash point.
"White Rose" kerosene (B)	118° F.
O. and C. test	129° F.
Ditto after shaking with—	
(*a*) 25% of its weight of clay	130° F.
(*b*) 25% " " coal	131° F.
(*c*) 5% " " animal charcoal	125° F.

The larger rises thus obtained practically proved that selective absorption of some volatile constituent had taken place. Further experiments were made in order to endeavour to discover the nature of this volatile constituent.

A sample of the kerosene was poured into another vessel and back some half-dozen times. The "tossed" kerosene so obtained was tested :—

Kerosene (B) "tossed"	Flash point 130° F.

The ease with which the volatile constituent was removed indicated that it was either gaseous or a liquid of low boiling point. In the former case the Engler drop point compared with the kerosene as bought should be unchanged ; in the latter case it should be raised. Actual trial gave the following figures :—

	Engler drop °C.	Calculated flash °F.
Original kerosene (B)	171 ..	129
Ditto after clay	172 ..	130
Ditto after "tossing"	171 ..	129

The drop point thus remained unchanged, indicating that the low flash constituent is gaseous in nature. In the second column above we give the flash points calculated from the observed drop points, using the Ormandy-Craven formula :—

$$\text{Flash point °K} = \text{Initial boiling point °K} \times 0{\cdot}736.$$

It will be noticed that the calculated figures are in agreement with those observed on the material from which the low flash constituents have been removed either by "tossing" or absorption.

Since the flash so calculated corresponds to the liquid hydrocarbons present, it follows that the 12° F. lower flash of the original kerosene is due to constituents not condensible at tap water temperature.

Attempts were now made to effect the removal of the dissolved inflammable gas by the following methods :—

(i.) Low pressure.
(ii.) Heating.
(iii.) Drawing air through.

(i.) 80 c.c. of kerosene (B) was taken and submitted to a water pump vacuum (about 30 mm. Hg. pressure). Bubbles were evolved in the kerosene freely at first, but sluggishly after a few minutes. After about 20 minutes the flash point of the residual oil was taken :—

Vacuum extracted kerosene. Flash point 124° F.

(ii.) 250 c.c. were refluxed for 3½ hours. The residual kerosene was tested. Flash point 124° F.

Heating on water bath under reflux condenser gave similar result. These results show that the dissolved gases are very firmly held by the kerosene.

(iii.) Dried air was aspirated through 80 c.c. portions of the original kerosene (B) and the loss in weight measured. The The results with the rise in flash point obtained in each case are given below :—

Litres of air drawn through.	Loss % weight.	Rise of flash point. ° F.
1	0·031	5
4	0·070	9
6	0·094	10
10	0·126	12
15	0·148	15

Artificial mixtures of gases and volatile liquids were now made with " tossed " kerosene, and the effect on flash point noticed. In some cases the effect of finely divided solids was tried.

Coal Gas.—Kerosene absorbs coal gas readily, lowering its flash point from above 130° F. to below 70° F. Tests were made on a mixture having a flash point of 123° F. with clay, ignited clay, and animal charcoal. No rise in flash point was found.

Ethylene.—Prepared from pure ethyl alcohol by the action of phosphoric acid. The dried gas was readily absorbed by " tossed "

kerosene. The alteration in flash point with various additions by weight of ethylene added was found to be as follows :—

% wt. ethylene.	Lowering of flash point ° F.
0·01	6
0·02	13
0·03	18
0·04	20
0·05	25
0·06	29

The above results were obtained when continuous stirring was employed, as in all other tests. Much lower flash points were obtained when no stirring was done, showing that ethylene is evolved more readily under the latter conditions.

A solution of ethylene in kerosene (flash 130° F.) having a flash point below 73° F. after shaking with 40 per cent. of clay flashed at 121° F.

Similarly, a solution flashing at 112° F., showed :—

	Flash point.
After 20% clay	121° F.
„ 30% „	128° F.

With a similar kerosene-ethylene solution flashing at 115° F. :—

	Flash point.
After 16% Fuller's earth	123° F.
„ 20% „ „	127° F.
„ 30% „ „	127° F.

The same solution was tested in the glass apparatus and gave :—

O. and C. test .. .. Flash point 131° F.

The Engler drop point of the solution was determined and found to be 172° C., whence the calculated flash point is 130° F. The Engler drop of the kerosene before adding the ethylene was also 172° C.

Pentane.—To "tossed" kerosene of 134° F. flash was added various proportions by weight of commercial normal pentane, and the flash point of the mixture found :—

% Pentane.	Lowering of flash point ° F.
0·031	3
0·062	6
0·098	9
0·148	12
0·177	17
0·197	20

A kerosene-pentane mixture flashing at 120° F. was treated with clay, etc., and gave the following results :—

After 5% clay	120½° F.
„ 40% „	122½° F.
„ 5% animal charcoal	120½° F.

Very little absorption thus seems to take place. The slight rises were probably due to gases known to be in the added pentane.

A pentane-kerosene mixture was tested in the glass apparatus, and in the Abel, and the figures obtained were :—

Abel flash	122° F.
O. and C. flash	135° F.

The kerosene before adding the pentane showed :—

Abel flash	131° F.
O. and C. flash	135° F.

Petrol.—To a "tossed" kerosene small amounts of Shell petrol were added, and the following figures observed :—

% wt. petrol added.	Engler drop.	Flash point. Calculated.	Flash point. Observed. (Abel.)
0	166	120 ..	120
2½	144	93 ..	90
5	132	77 ..	74

Ether.—To kerosene small quantities of ethyl ether were added, and the following figures obtained :—

Ether % wt.	Flash °F.
0	125
0·067	120
0·134	115
0·268	106

A sample of "Shell-Mex" vaporising oil 0·784 S.G. was now examined, and found to contain a volatile absorbable fraction :—

	Original oil.	"Tossed" oil.
Abel flash point	82° F. ..	89° F.
O. and C. flash point	88° F. ..	90° F.

	Flash point.
After pumping out under vacuum	85° F.
„ distilling off 1 drop from 80 c.c. in vacuo ..	85° F.
„ „ „ 2 drops „ „ „ „ ..	86° F.
„ „ „ 5 „ „ „ „ ..	90° F.
„ shaking with 40% clay	86° F.

Air was drawn through in a manner similar to that with kerosene, and the following results obtained :—

Litres of air drawn through.	Loss % wt.	Rise in flash point ° F.
1	0·032	3
2	0·048	4
4	0·112	3
6	0·159	4
10	0·279	4
20	0·510	7

It would appear that about 4° F. rise is obtained quickly, and that no further rise in flash takes place until a considerable amount of lighter liquid fractions begin to evaporate.

Discussion and Summary.

It appears from the foregoing results that kerosenes are liable to contain small amounts of very volatile inflammable matter, probably gaseous in nature. In Fig. I., Curve "A" shows the rise in flash point of kerosene consequent on the removal of increasing proportions by weight of the kerosene by air blowing. It will be noticed that the effect of the removed material lies between the

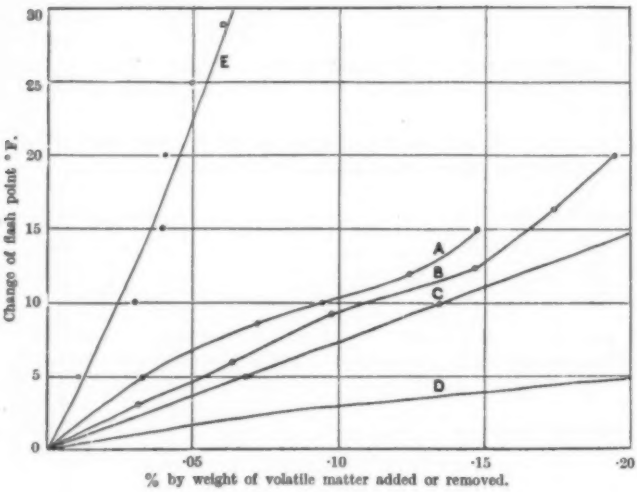


Fig. I.

A. Rise of flash point of kerosene (B) by air bubbling.
B. Lowering of flash point of kerosene by pentane.
C. Ditto by ether.
D. Ditto by petrol spirit.
E. Ditto by ethylene.

corresponding effects for ethylene and pentane, which indicates that the bulk of the removed material is gaseous at room temperature. Moreover, the material removed by air blowing must consist partly of heavier liquid bodies, so that the effect of the gaseous material is not fully apparent from the curve.

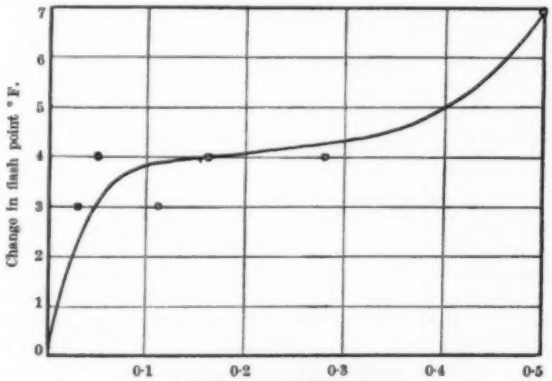
In Fig. II., which gives a similar curve for vaporising oil, it will be noticed that a rise in flash of 3—4° F. occurs very quickly, and that much more material has to be removed before a further rise of 1° F. takes place. This, again, would appear to indicate the presence of a gaseous, or, at any rate, exceedingly volatile body.

The rise in flash of kerosene on shaking with inorganic solids is of the same order as that produced by coal. The maximum rises by absorption found correspond very fairly with the difference between the Abel and the O. and C. flash points, as shown in the following tables :—

	(1)	(2)
Kerosene (A)	2 ..	0
,, (B)	13 ..	11
,, and ethylene	15 ..	16
,, ,, pentane	2 ..	4
Vaporising oil	4 ..	6

(1) = mean change of flash point by absorption with solids.
(2) = difference between Abel and O. and C. flash. (In the case of kerosene-pentane the difference before adding pentane is given.)

In each case tried, moreover, the flash point, as calculated from the Engler drop point, agrees with the O. and C. flash. As would be expected, and as shown with ethylene, the presence of small quantities of gas has no effect on the Engler drop nor the O. and C. flash point. Hence the difference between the two flash points, or



Rise in flash point of vaporising oil by air blowing.
FIG. II.

between the Abel flash and that calculated from the drop point is the measure of the gaseous or extremely volatile matter present.

Practically no absorption took place in mixtures containing either coal gas or pentane, but was very marked in the case of added ethylene or the volatile constituent of kerosene (B). It may, therefore, be fairly assumed that such absorption and consequent rise of flash point can only be expected when the volatile body is of an unsaturated nature. Hence the rise in flash by absorption will not correspond to the difference between the Abel and the O. and C. flashes when saturated volatile bodies are present, though the difference between the two flashes will correctly indicate the presence of the volatile constituent. Thus, with kerosene-pentane we have :—

Lowering of flash by adding pentane	12° F.
Difference of Abel and O. and C. flash	13° F.
Raising of flash by absorption	2° F.

In general, therefore, it has been shown that traces of abnormally volatile bodies occur in kerosenes, and that an idea of their amount may be gained from the difference between the Abel and the O. and C. flash points. It is shown also that the very volatile bodies in kerosene are largely absorbed by clay or animal charcoal, and are probably of an unsaturated nature.

The authors desire to express their thanks to Dr. W. R. Ormandy for his interest and help in the above work, and also to the Fuel Research Board of the Department of Scientific and Industrial Research, and to the Salters Institute of Industrial Chemistry for financial assistance.

CORRESPONDENCE.

Kukkersite.

MR. E. H. CUNNINGHAM CRAIG'S replies to discussion (J. 32, p. 349) :—

In reply to the written contribution from Mr. A. Beeby Thompson, there are several points which must be mentioned.

There need be no fear as to great variation in the quality of the shale ; the deposit is remarkably uniform over a very wide area, though the amount of comminuted shell fragments varies in different localities. The tests made have not been of carefully selected material, but of the ordinary run of the seam. The limestone bands are easily picked out as they naturally separate themselves from the shale : during the writer's visit to Esthonia the cost to the Government of the excavation by manual labour,

including hand-picking, loading on trolleys, pushing trolleys to siding and loading on trucks, worked out at 10½d. per ton.

As regards ideas of accumulation of oil Mr. Thompson seems to have confused the phenomena of the migration of a crude oil with the phenomena of the adsorption of the heavy fractions of an inspissated oil by colloidal organic matter. Surface tension causes such adsorption, which cannot be resolved again by incursion of water. Adsorption being proportional to surface, there can be very little in the coarser sediments compared with that in colloidal argillaceous beds. In coarser sediments which allow circulation of water, oil, unless very highly inspissated, can be completely replaced by water. In zones nearer the surface, and therefore at a lower temperature, the surface tension is greater, and a colloidal bed can adsorb an oil which water could easily remove from a sandstone.

It has been demonstrated frequently in the last few years that the difference between a shale in an oilfield impregnated with crude oil and the most highly inspissated oil-shale is merely one of degree and not of kind: every gradation between the two can be demonstrated, and Dr. David T. Day, in the *Handbook of the Petroleum Industry*, makes this quite clear.

In answer to Professor Madgwick, the writer would point out that the question really resolves itself into this: "Is an oil shale due to impregnation by oil or to matter, not petroleum, originally contained in the deposit?" Mr. Murray Stewart's theory is simply the theory of colloidal adsorption, stated somewhat differently. The word "sapropel" is as dead as "Kerogen," for the idea entails an assumption; the word "Kerogen" was useful as expressing a fact, before the full significance of the fact was understood. Dr. David T. Day gives in *The Handbook of the Petroleum Industry* a graph dealing with percentages of volatile and ash in coals, lignites, torbanites, oil shales and artificially-formed oil shales, which points conclusively to oil shales having been formed by impregnation.

In answer to Mr. Romanes, the writer would suggest that he has hardly appreciated the significance of the evidence.

A manjak vein is conclusive evidence of the intrusion of petroleum from below. Quite apart from such intrusion, migration of oil can be proved to have taken place in many countries through hundreds of feet of strata, leaving little or no trace except in such beds as have sufficient colloidal content—*e.g.*, to a small extent the Dictyonema shale and to a very much greater extent the "Kukkersite." The special simple conditions existing in Esthonia must not be forgotten; the waterlogging of the sandstones, and the gradual transference of oil upwards, inspissating as it went, hardly require explanation in the circumstances.

REVIEWS.

"Oil Flow in Pipe Lines." By R. S. Danforth.

The problem of the measurement of viscosity is of increasing importance, especially to the petroleum industry, and the interesting series of tables prepared by Mr. R. S. Danforth, which is now before us, is a valuable contribution to the literature of the subject.

From an engineering point of view the present methods of viscosity determination of petroleum products are not at all satisfactory. Not only are there in use a number of different instruments—Redwood, Saybolt, Engler, etc.—but the results obtained by them are by no means easily comparable and are not capable of being reduced to absolute viscosity figures with any degree of accuracy. It is therefore to be hoped that in the near future an International Congress will endeavour to get one type of viscometer adopted in all countries. It will then have to be debated whether any one of the above-named instruments is really sufficiently accurate for modern requirements, more especially when dealing with the very viscous oils that are now on the market. In France the Barbey "Ixometre"—that measures the fluidity and not the viscosity—is generally used, and this instrument has a great advantage over the viscometers mentioned in that the head of oil is constant, the only variable being the quantity of liquid that passes through the instrument under constant head and in a definite period of time. This instrument was originally designed for dealing with lubricating oils, and is, unfortunately, not especially suitable for dealing with extremely viscous oils.

We sometimes wonder whether an instrument on the lines of Dr. Searle's viscometer would not give more accurate results in practice than any of the instruments at present in use. All engineers who have to deal with oil pumping problems will, however, be very grateful to Mr. Danforth for the great trouble he has taken to get out these highly interesting and valuable charts. It is, of course, obvious—and this is in no way derogatory to Mr. Danforth's work—that a great deal of practical experience of oil pumping is required to make the best use of these charts. It is not clear to us whether Mr. Danforth intends them to be used for calculations appertaining to oils of a definite origin, for we venture to think that they are not applicable to all oils, owing to the fact that all oils have not the same viscosity characteristic.

It should further be noted that Mr. Danforth bases his viscosity conversion factors on approximate formulæ given by Mr. W. H. Herschel, but it should have been stated that these factors are only applicable for readings over 70 seconds Redwood No. 2 and Saybolt Furol, and over 400 seconds Engler, Saybolt Universal and Redwood No. 1, and for over 10 degrees Engler when the readings of this viscometer are so converted, because it is at these points that the conversion curves for the different standards become parallel straight lines.

It would be most interesting to have Mr. Danforth's remarks on these points.

In this country the conversion factor for Redwood No. 2 and Redwood No. 1 is taken as 10, but in America it appears to be taken as 9·2—see chart E.

G. W. E. Gibson.

"The Economics of Petroleum." By Joseph E. Pogue.

The author's purpose in writing this book was "to present, in perspective, the more important economic facts relating to petroleum; to interpret the changes that are rapidly taking place in this field; and to project the trend

of these changes into the immediate future." "The prime function of a science is to enable us to anticipate the future in the field with which it has to deal."

The scope of the book is practically limited to the United States of America but as over 60% of the world's output comes from that country, and as the expansion and development of the petroleum industry have been so phenomenal there, the results obtained from a study of the economic conditions there may be taken as generally applicable elsewhere, the time factor only requiring modification.

The book opens with a chapter on the economic organisation of the industry. This is followed by three chapters on the trend of oil production and the available resources. The rapid approach of the production of the United States to its peak, and the widening gap between production and consumption, with its economic consequences, is emphasised. Estimates of the possible productions of many countries which are hardly yet explored are given. Many geologists will hardly agree that "while such figures are doubtless highly speculative" they are "reasonably satisfactory as a basis of discussion."

Several chapters follow, dealing with the transport of crude petroleum, the trend of refinery practice and the outlook for oil refining. The refinery capacity of the United States is evidently far in excess of its needs. This does not, however, apply to other countries. Six very interesting chapters deal with individual products. The relation of quality of products and of modifications in refinery practice dependent on changes in relative demand are well worked out. The latter half of the book deals with the prices of petroleum and its products, the relation between price and production. The motor fuel problem is also discussed in all its phases. The last few chapters deal with the relation of the coal and the shale industries to the petroleum industry. The book concludes with chapters on the need for economical use of petroleum products, the crime of using such valuable products largely as mere fuels being emphasised, and on the use of statistics in the industry.

The book is exceedingly well illustrated by very numerous graphs explanatory of all the points discussed. The graphs are usually supplemented by tables of the data from which they have been drawn up. The author does not claim to give any technical account of the industry beyond what is necessary to enable the non-technical man to follow the argument. This is done in a very lucid way.

The book is well printed and bound, free from misprints, and although expensive (33s.) is well worth careful study by anyone interested in the petroleum industry.

JAMES KEWLEY.

"GAS CHEMISTS' HANDBOOK." Compiled by the Chemical Committee, Technical Section of the American Gas Association. March 1, 1922. Second Edition. Published by the American Gas Association, 130, East Fifteenth Street, New York.

This is a useful handbook of analytical methods applicable in gasworks, which have been accepted by an Editorial Committee of the American Gas Association, as representing current practice among leading gas chemists in the United States. The Chairman of this Editorial Committee of three members, Mr. E. C. Uhlig, is himself responsible for four sections of the text; one of his colleagues, Mr. O. A. Morhous, writes three other sections, and the third member, Mr. C. C. Tutwiler, writes one. The remaining ten sections in the volume have been contributed by eight other authors, with the aid of a number of collaborators. Having regard to the auspices under which the handbook has been prepared, the methods of analysis described may be regarded as having received semi-official recognition as the standard American methods at the present day.

The Editorial Committee and authors are to be warmly congratulated on the manner in which they have done their work. The matter is well arranged; the descriptions of methods are as concise as is consistent with precision and lucidity, and, generally speaking, the methods are substantially those which are now accepted in this country.

Only four sections of the volume have any very direct interest to chemists engaged in the mineral oil industries. These sections deal with gas oil; light oils and light oil products; tar from water gas and coal gas; and lubricants. The cracking test, as applied to gas oil on gas works, is here described in as convenient a form as can well be conceived for small laboratories; and, in order that the cracking may take place in the presence of water-gas, even if there is no works' supply of blue water gas available, accessory laboratory apparatus for producing blue water gas is included in the description. But just as different works and different operators will obtain somewhat different results from the same oil when cracked in carburetted water-gas plant, so the results of a laboratory cracking test of oil are hardly likely to be identical in different places and in different hands, even if the apparatus and procedure are apparently identical. Hence it is an advantage to make comparative cracking tests from time to time of a standard grade of gas oil, and the American Gas Association would do good service by arranging that one or more such oils should be obtainable in single drums, under seal with a guarantee of identity. The cracking test for ascertaining the value of an oil for carburetting water-gas intended for public gas supplies obviously must differ in principle from a cracking test intended to ascertain the potentialities of a heavy mineral oil as a source of cracked motor-spirit.

The testing of road tars for viscosity or consistency has developed here on different lines from those obtaining in the United States. The Engler type of viscosimeter was discarded in this country after trial in the early years of road-tarring, and the float test has never come into favour here, though both continue to be used in America. On the other hand, the Hutchinson viscosity gauge or consistometer, which is of the "sinker" type, has become accepted as a standard instrument in Great Britain, and has proved invaluable in the grading of road-tars and other bituminous road dressings. It is a pity that instruments of this class, which are of great utility in industry, are commonly of different patterns in this country and in the United States, with the consequence that many technical observations and reports have little significance outside their country of origin. In the case of tars, pitches and bitumens, which contain insoluble matter in admixture with the liquefiable hydrocarbon matter, there can be no true viscosity susceptible of measurement on a scientific basis, and comparisons become possible only if there is agreement as to a conventional or standard method of testing. Such agreement is sorely needed between England and America in regard to the apparatus and procedure for comparing the consistency of tars, pitches, asphaltic bitumens and the like. Perhaps the Roads Department of the Ministry of Transport here and the Office of Public Roads in the United States will ultimately agree upon standard methods for the empirical tests of the binding and surfacing materials employed by the road engineer.

W. J. A. BUTTERFIELD.

"HANDBOOK OF THE PETROLEUM INDUSTRY." By David T. Day and Others.

GEOLOGICAL PART.

The main feature of this book is the very broad and comprehensive view taken of the subjects with which it deals. The authors are obviously *au fait* with all geological data relating to petroleum in the United States, and as the United States produces two-thirds of the world's oil supply, there is some justification for entitling the book, "A Handbook of the Petroleum Industry."

It is, perhaps, unfortunate that the attempt has been made to deal with the oil industry of the world, as much of the evidence relating to other countries has obviously been compiled, and not obtained at first hand by the authors. Consequently, we find in accounts of European, Asiatic, and African oilfields a great deal of very sketchy description, and frequently statements that are entirely inaccurate. This is obviously not the fault of the authors, but of the sources from which they have obtained much of their information. Even the geographical information is not always correct, and confusion between Arabia, Palestine, and Turkey, has, unfortunately, crept in; while placing Bahrein in the Red Sea instead of the Gulf of Persia, is hardly excusable.

As a book of reference for anyone wishing to study the oilfields of the United States, the book will be invaluable, but on phenomena, *e.g.*, mud volcanoes, stated not to occur in the United States, the authors' information is somewhat defective.

There are very interesting sections upon the methods used in the more mechanical forms of geological field-work, especially as applied to the mapping of geological formations of low dip, and in open country.

On the subject of Oilshales a very comprehensive view is taken, the term being used to include lignites, torbanites, cannels, asphaltites, and even coals as well as every form of true oilshale. Possibly this serves to obscure some of the most interesting scientific points, but it has certainly a practical bearing on industry, and this is a book on the Petroleum Industry. The description of retorting methods for oilshales and the ideals to be realised in modern retorting processes are admirably reviewed and set forth, but as the author states, the technology of oilshale, so far as the United States is concerned, is "in a formative stage," and hence he writes the chapter "with the full realisation of its limitations and its incompleteness." Despite this incompleteness, the chapter on Oilshale is a very valuable one, and should be studied by every technologist interested in the subject.

This is emphatically a book to read, to study, and to keep for reference.

C. H. C. C.

Chemical Part.

The well-known treatises of Engler and Redwood might well be supposed to fill completely any gaps that existed in our knowledge of naphthology, but it can be said, in all fairness, that Dr. Day's massive treatise is in no way redundant, and that in point of fact, it gives a fresh presentment of the subject as a whole. Indeed, it would have been a matter of no little comment had our American cousins not given to the world an authoritative work dealing with an industry so predominatingly their own. A healthy spirit of co-operation is evidenced in a preliminary glance through the 2000 pages of closely printed matter, and the editor has had placed at his disposal the collective ability of no less than fifteen experts. From the point of view of the chemist, the chief sections are :—

(1) The characteristics of petroleum by D. T. Day, comprising a very comprehensive set of data relative to the chief groups of hydrocarbons, oxygen, sulphur, nitrogen compounds, important physical properties and details of the nature of the chief American Crude oils.
(2) Petroleum Testing methods, by T. D. Delbridge, an exceedingly useful compilation, extending to 140 pages, and a veritable manual of American procedure.
(3) Natural Gas Gasoline, by H. C. Cooper.
(4) Asphalt, by R. G. Smith.
(5) Oil Shales, by D. E. Day.
(6) Refining, by A. D. Smith.

(7) Cracking Processes, by R. B. Day.
(8) Fuel Oils, by W. N. Best.
(9) Lubrication, by J. D. Gill.

Detailed criticism of so voluminous a work would be of little value, but there is no doubt as to the thoroughness and care which have been exercised in the compilation. One point, however, might be referred to, that is, the absence of references to the work of chemists other than American. Thus there is no allusion in the index to Redwood beyond viscometry, or to Engler excepting his flask and viscometer, to Friedmann, to Gurwitsch, or to Hardy, among other well-known names. These omissions to extra-American activities are to be deplored in that they militate against that catholicity of outlook, which should distinguish men of science. A. E. D.

"The Petroleum and Allied Industries." By James Kewley, M.A., F.I.C., F.C.S. Baillière, Tindall and Cox, London.

Mr. Kewley's manual has been written in the hope that it will appeal to the many non-technical workers in the petroleum industry. In our opinion the book will go much further than the author's very modest intention, because it is not only a well-written scientific exposé of the subject, but also it is singularly comprehensive. As an introductory text-book to a course of lectures, we can imagine nothing more useful, whilst the general reader will find a clear, brief and explicit development of elementary naphthology. The Introductory Section is concerned with terminology, the chemistry, geology and origin of petroleum. A section on the application and uses of Natural Gas is followed by an account of the drilling, storage and refining of crude oil. The shale oil industry is succinctly described, and a variety of tars are discussed. The natural solid and semi-solid bitumens, with their uses as road-making materials, form the subject of a special chapter, whilst the final section is devoted to the character and application of petroleum products. A summary of the chief testing method is appended. Each section is provided with a list of reference books.

The book is excellently finished and illustrated, and can be very highly recommended. A. E. D.

"The Chemistry of the Non-Benzenoid Hydrocarbons." By B. T. Brooks, Ph.D. Chemical Catalogue Co., New York.

It is with considerable pleasure that we welcome this valuable contribution to Applied Organic Chemistry, written by an American member of the Institution of Petroleum Technologists. It has become a reviewer's cliché to state that a book fills a long-felt want; but in all truth, Dr. Brooks has written a pioneering work, and has struck out for himself a path which one can only wonder was not trodden long ago. The student of Organic Chemistry very soon is attracted to the Aromatic section of his work, partly because of its intrinsic interest, partly because of the wealth of discovery that has lain therein—dyes, drugs, perfumes and explosives—and partly because his text-books naturally trend thereto. Dr. Brooks has attempted to—and has succeeded in—demonstrating that the aliphatic and cycloparaffinoid compounds also have industrial and scientific importance, and he has marshalled in a convincing way the various groups of non-benzenoid derivatives. A brief idea of the scope of the work may be gained from the following summary: paraffins and their properties, the ethylene bond, polymerisation of hydrocarbons, cyclic non-benzenoid hydrocarbons, the methanes, bi and tri cyclic compounds, the camphor group, rearrangements and physical properties. The author's well-known standing as a petroleum chemist has led him to devote a large amount of space to the chemical side of naphthology, and to all members interested in this subject the book can be unreservedly recommended. The printing and get-up are admirable. A. E. D.

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AMERICAN CHEMICAL SOCIETY.

PETROLEUM SECTION.

Abstract of papers submitted at the Pittsburg Meeting, Sept., 1922.

1. THE VAPOUR PRESSURE OF SOLUTIONS OF BENZOL, HEXANE AND CYCLOHEXANE IN LUBRICATING, GAS, ABSORBENT AND VEGETABLE OILS WITH MOLECULAR WEIGHT DATA.—Robert E. Wilson and Edward F. Walls, Mass. Inst. Technology, Cambridge, Mass.

A knowledge of the vapour pressure of volatile solvents, such as benzol and hexane, dissolved in various types of relatively non-volatile oils, is of commercial importance in a considerable number of applications. Of these, the three most important are: (*a*) the dilution of crank-case oils with fuels in internal-combustion engines; (*b*) the complete removal of volatile solvents from oils and fats obtained by the solvent extraction processes; and (*c*) the recovery of solvents by scrubbing with cold absorbent oils, as in benzol recovery from coal gas.

In spite of the commercial importance of such information, a very little work has been done along those lines, even at ordinary temperatures, and no generalisations have been drawn which would make it possible to calculate approximate figures for a specific case. It was the object of the work described in this paper to make possible such generalisations and approximate calculations for the three general types of volatile hydrocarbons—paraffin, naphthene, and aromatic—dissolved in most ordinary types of oils. Molecular weights were determined on all of the oils used, and the amount of vapour pressure lowering for different combinations was compared with the theoretical as predicted by Raoult's law. It appears that the amount of lowering produced by different types of combinations is fairly definitely predictable, and that the results for any combination met with in practice can be calculated with quite satisfactory accuracy for any ordinary temperature if the vapour pressure of the pure volatile constituent is accurately known, together with the molecular weight and type of hydrocarbons present in the oil.

Molecular weight data included in the article are also of interest in being the first published data on a fairly wide range of the higher mineral oil fractions, the results having been obtained by measuring the freezing point lowering in benzol.

2. FURTHER DATA ON THE MEASUREMENT OF THE EFFECTIVE VOLATILITY OF MOTOR FUELS.—Robert E. Wilson and Daniel P. Barnard, 4th. Mass. Inst. of Technology, Cambridge, Mass.

This paper is an extension of the work presented at the Rochester Section, where some new methods of determining the effective volatility were described, and the results on three typical fuels presented in some detail. This work has been extended to more than a dozen fuels in this laboratory, and this, combined with data obtained in other laboratories, makes it possible to draw certain generalisations as to the effect of the shape of the distillation curve on the effective volatility of the fuel in so far as it affects the completeness of vaporisation and proper distribution in the manifold.

In the light of these investigations, the writers believe that a single specification for the 85% point should replace all the present limitations at 50, 90 and 96%, and similarly that the effective starting volatility can best be measured as a routine proposition by a specification at the 25% point rather than at the 5% or initial point.

3. Some Applications of Vapour Pressure Measurements.—Harold S. Davis and A. D. Little, Inc., Boston.

An improved differential-pressure apparatus for measuring vapour pressure is described. The apparatus can be successfully applied to the control of light oil recovery plants. Actual data from plant tests are given.

It has been suggested that oils could be standardised for efficiency in gas absorption by preparing solutions containing the same concentration of benzene and measuring their vapour tensions. Experiments on seven representative absorbent oils have shown no appreciable differences between them in this respect, and practical variations in their efficiencies must be attributed to other factors rather than to their ability to lower the vapour tensions of dissolved benzene.

Some applications of vapour-pressure measurements in gasoline recovery are discussed.

4. A Rapid Test for the Decolorising Efficiency of Fuller's Earth.—Leon W. Parsons and Leonard R. Churchill. Mass. Inst. Technology, Cambridge.

The paper covers the development of a rapid test for determining the relative efficiencies of various samples of Fuller's earth for decolorising purposes. This test was designed to avoid the necessity for resorting to tedious filtration processes in the laboratory to determine the quality of the clay, and it serves two purposes; first, to compare the decolorising power of various clays used in experimental investigations; and, second, to serve as a rapid control test for plant operation. A survey of the properties of Fuller's earth which would parallel its power to decolorise oils is given, and the development of several different tests is indicated. The most satisfactory test, however, is based on the reaction between Fuller's earth and pinene. Fuller's earth reacts vigorously with pinene to form polymerised products with the development of a very large amount of heat. The amount of temperature rise is used as a measure of the efficiency of the clays, and data are given to show the relation between this pinene efficiency and the decolorising power of such clays. A discussion of the mechanism of the reaction, the materials used, and the application of the test for laboratory and plant use is indicated.

5. Effect of Certain Physical and Chemical Properties of Emulsifying Agents on the Stability of Oil-Water Emulsions.—Leon W. Parsons and Brian Mead. Mass. Inst. Technology, Cambridge, Mass.

This paper embodies further studies on Nujol-water emulsions, and a comparison of these systems and commercial mineral oil emulsions. The technique of investigating these emulsions, making use of microphotographic apparatus and the ultra-microscopic, is given. A study has been made of the following factors of extreme importance in connection with the stability of emulsions: Physical characteristics of emulsifying agents; effect of opposing emulsifying agents in the same system; action of oil soluble colloids on the emulsifying agents; and similarity between behaviour of soaps and of finely divided solids. The importance

of relative wetting of the different phases by the emulsifying agent is emphasised, and it has been found that several common emulsifying agents give abnormal results with regard to type of emulsions produced. This latter observation has important bearing on the mechanism of the behaviour of soaps and finely-divided solids as emulsifying agents. Certain parallelisms have been observed between the behaviour of the emulsifying agents used for Nujol-water systems and those present in commercial oil emulsions.

6. Engine Experiment with High Sulphur Fuels.—Carl O. Johns, Standard Oil Company of New Jersey.

These experiments were conducted by Professor George Winchester, formerly in charge of the Engine Research Laboratory of this company. A Hvid type of Diesel engine ("Thermoil") was used. Several fuels were tested to ascertain the effect of both free and organic sulphur. The runs were continued for 384 hours. The fuels included high sulphur Mexican crude and gas oil, and mixtures of these with a polymer oil containing about 19% of combined sulphur, or 10% of sulphur in the mixed fuel. The running time for these fuels was 180 hours. The compression in the engine reached 500 lbs. A run of 204 hours' duration was made with kerosene, containing 0·85% of free sulphur in solution. Frequent examinations of the dismantled engine disclosed no evidences of corrosion, nor was the copper tubing affected appreciably. Trouble was encountered by the excessive formation of carbon which deposited on the exhaust valve and clogged the spray holes to the cylinders.

7. Gasoline Saved on Government Trucks, Due to Adjusting the Carburettors by Gas Analysis.—G. W. Jones and A. C. Fieldner. Bureau of Mines, Pittsburgh, Penn.

Carburettor adjustments by exhaust gas analysis on 15 trucks, varying from 2½ to 7½ ton capacity, showed that all but one were too richly adjusted for maximum power and economy.

The adjustments made gave an increase of over 20% in mileage for the first month after testing, and 16% increase for the second month.

The CO_2 indicator used for making the adjustments while the trucks were operated was found to be practical and easily operated by the layman inexperienced in the handling of chemical apparatus.

An analysis of the exhaust gas under the conditions in which the truck is operated gives an absolute indication of the carburettor adjustment, and removes all guesswork.

By adjustment for maximum power and economy is meant the leanest adjustment without reduction of power.

8. Some Factors Affecting the Accuracy of Saybolt Viscosity Measurement and their Control.—P. E. Klopsteg and W. H. Stannard, 460, East Ohio Street, Chicago, Ill.

Without a suitable form of automatic temperature control, it is impossible to secure experimental conditions which conform to A.S.T.M. specifications, namely, that "The bath shall be held constant within 0·25° F. (0·14° C.) at such a temperature as will maintain the desired temperature in the standard oil tube." At any rate, the requirements cannot be met with the form of bath described in the specifications.

In this paper the development is described of an electric heating and regulating device which, at a temperature of 212° F. (100° C.) will hold

the bath temperature constant within 0·1° F. (·05° C.) throughout an entire day's run without attention.

Conditions within the oil tube are also considered, and it is shown that when the A.S.T.M. method of stirring is followed, even with the bath temperature very constant at 212° F., variations of 0·5° F. are observable on a sufficiently sensitive stirring thermometer. A method of stirring is then described by means of which it was found possible to hold the temperature of the sample under test constant to 0·01° F. over a period of many minutes. Tabulated data show the Saybolt readings at different temperatures, for samples tested under "standard" conditions, and with the new apparatus.

9. CRACKING OF UNSATURATED HYDROCARBONS BY HEAT TO GIVE SATURATED HYDROCARBONS.—Ralph H. McKee and Ralph T. Goodwin, Department of Chemical Engineering, Columbia University, N.Y.

The real liquid fuel reserve of America is in its oil shale deposits. In a study of oil shale we have found that distillation in vacuum gives an intermediate product which, on heating, goes over into petroleum. A study of this intermediate from various oil shales shows it always to be highly unsaturated (dissolved by 66° Bé sulphuric), sometimes 100% unsaturated. If this waxy intermediate is distilled at atmospheric pressure, there is obtained a petroleum containing 48% saturated hydrocarbons. Redistillation brings about additional cracking with a further increase of the percentage of saturated hydrocarbons.

Ordinarily we expect the cracking of hydrocarbons to proceed with increase of unsaturated constituents; but here we have the reverse; the unsaturated constituents decreased 48% on the first distillation, and still more on redistillation.

10. THE REFINING OF GASOLINE AND KEROSENE BY HYPOCHLORITE SOLUTIONS.—A. E. Dunstan and B. T. Brooks, Anglo-Persian Oil Company and Mathieson Alkali Works.

The present paper is a description of the process developed and perfected by A. E. Dunstan and his associates in the Anglo-Persian Oil Company. It is the first successful industrial utilisation of hypochlorite solutions for refining of petroleum oils. Gasoline and kerosene may be refined in this way with treating losses of less than one-half of 1%. Malodorous compounds, including sulphur derivities, are removed. The treated oil is free from chlorine, is free from substances which develop acidity, gummy substances or discolorations on standing, and it eliminates obnoxious waste products, such as acid tar, eliminates acid recovery, and the process can be carried out in the usual form of treating apparatus. The new method, therefore, is cleaner and easier to operate, gives a much better refined product, and the refining costs are markedly lower, particularly when the losses incidental to sulphuric acid refining are included in the comparison. The process is particularly adapted to light distillates from high sulphur crudes, and the savings are greatest in the case of cracked gasolines.

11. REFINING OF CRACKED HYDROCARBON OILS.—Jacque C. Morrell and Gustav Egloff.

The various methods of refining light petroleum distillates are discussed. The general methods involved are: (*a*) Physical; (*b*) Chemical. The physical method of refining involving the use of various adsorbent

earths, though giving good results, cannot compete commercially with chemical methods of refining. The chief chemical method in use to-day is the sulphuric acid method of refining, and it is the use of various modifications of this method upon which the authors lay the greatest stress. Applying various modifications of this method with regard to subsequent treatment, as well as concentration and amounts of reagents, the authors have successfully refined cracked distillates from the following raw materials, and have produced water-white gasoline of sweet odour, as well as a product which will pass the corrosion and doctor test: (*a*) California gas oil; (*b*) Mid-Continent fuel oil; (*c*) Mid-Continent gas oil; (*d*) Mexican gas oil; (*e*) Texas fuel oil. The raw oils were cracked on a commercial scale in the gas-liquid phase. The authors describe in detail the methods used with full discussion of the theories involved, especial stress being laid upon the treatment of hydrocarbons containing sulphur compounds. The effect of the sulphuric acid treatment upon the unsaturate per cent. is also discussed. It is concluded that, following the methods and principles laid down by the authors, all types of cracked hydrocarbons, regardless of the type of raw oil used to produce them, can be successfully treated to produce water-white products of sweet odour.

12. Fractional Distillation of Various Petroleums under Reduced Pressure with New Type Receiver.—Jacque G. Morell and Gustav Egloff.

A new type of receiver for fractional distillation under reduced pressure is described. The general principles and elements of the receiver are as follows:—The receiver is made up of two graduated chambers, the whole being arranged so that it can be properly mounted. These chambers are connected by a stop-cock. The arrangement is such that one fraction may be drawn off from the lower chamber while the next fraction is being distilled in the upper chamber. The rate of distillation is always under perfect control. With regard to the fractional distillation of petroleums under reduced pressure, following a general discussion of the theory and experimental method, comparative distillations of the following crude oils under atmospheric distillatic and vacuum distillation are shown: Kentucky Crude, Arkansas Crude, Colorado Crude, Ohio Crude, California Crude, Texas Crude and Mexican Crude. A discussion of the results from the viewpoint of unsaturated content, percentage of paraffin, and viscosity of lubricating stock for both types of distillation follows the experimental results.

13. Sulfo-Acid Bodies in Lubricating Oils.—G. L. Oliensis. Barber Asphalt Company, Madison, Ill.

When lubricating oils are over-treated or prematurely neutralised in the agitator, some sulfo-acid compounds from the sludge are reincorporated in the oil. The breaking of the resultant emulsion or blowing of the oil to brightness causes some of these compounds to go into perfect solution in the oil. The Conradson Demulsibility and Vacuum Company steam tests then cause these bodies to precipitate as a soapy layer below the oil, thus constituting qualitative tests for such material. Evidence indicates that a foamy layer differs from a homogeneous layer only in concentration. It is indicated that this layer is caused by salts of inorganic acids, etc. The significance of the presence of such material and the usefulness of the Conradson test for its recognition are discussed.

SYMPOSIUM ON "LUBRICATION FROM THE CHEMISTS' VIEW-POINT."

14. LUBRICANT AND ASPHALTIC HYDROCARBONS IN PETROLEUM.—C. F. Mabery, Case School of Applied Science, Cleveland.

The work of this paper is a continuation and expansion of the preliminary notice presented at the Rochester meeting, in which it was stated that an investigation had been begun to ascertain the composition of the part of petroleum that cannot be distilled without decomposition.

This work is now well advanced, and a large number of hydrocarbons have been identified in typical West Virginia, Pennsylvania, Mecca, Texas and Baku oils. The more soluble lubricant hydrocarbons, at least those of lower molecular weight, collect for the greater part in the D series (more soluble) and the heavier asphalts in the H series (less soluble).

The two series differ widely in viscosity and in lubricant quality. Tested on a frictional machine, the D-s are superior in wearing quality to the H-s. The highest viscosity is shown by the Mecca D- No. 8, the last one of the series, $C_{72}H_{132}$, 5461 seconds, in an Ostwald tube, water standard 0·25 S. at 38 C., 100° F., 2780 Saybolt.

This paper also gives tables of viscosity, iodine numbers, showing unsaturated hydrocarbons, and values by the formolite reaction showing internal cyclic unsaturation.

15. VISCOSITY TEMPERATURE CURVES OF FRACTIONS OF TYPICAL AMERICAN CRUDE OILS—SECOND PAPER.—F. W. Lane and E. W. Dean, Bureau of Mines, Washington, D.C.

The present paper contains new viscosity-temperature data on oils, derived from typical Mid-Continent and Gulf Coast crudes. Similar data, which were previously presented for fractions distilled from typical Pennsylvania, Wyoming, and California crude oils, are given in revised form. This completes the investigation undertaken by the Bureau of Mines on the viscosity-temperature relations of oils, and it is believed that the figures cover a fairly wide range of petroleum fractions of known origin and accurately determined physical properties.

It has been shown that the relation between the kinematic viscosity and the temperature may be represented by the equation:

$$V_k = \frac{1}{K + At + Bt^2}$$

in which V_k is the kinematic viscosity, t is the temperature in degrees centigrade, and K, A and B are constants characteristic of each oil. The relationship between kinematic viscosity and temperature may also be represented by the equation: $V_k = C(t+B)^n$, in which V_k and t have the same significance as before, and where C, B, and n are constants characteristic of each fraction. In its logarithmic form this equation becomes: $\log V_k = \log c + n \log (t+B)$. When B is suitably chosen, and the equation is used in its logarithmic form, the viscosity-temperature relationship for all fractions (including gasoline, kerosene, gas oil and lubricants) may be represented by straight lines. In the case of lubricating oils derived from the same crude, these straight lines converge for all practical purposes to a single point. This common point is somewhat differently located for oils produced from different crudes. Thus, if the source of a lubricant is known, the viscosity-temperature relationship for oils derived from the same crude may be determined when the viscosity at one suitable temperature and the value of B are available.

The possibility of extending this principle to apply to all oils of whatever source is discussed briefly and illustrated with appropriate data.

Certain preliminary experiments have also been undertaken on the relative effect on the viscosity of oils derived from a given crude of : (1) treating the crude oil with concentrated sulphuric acid ; and (2) filtering the crude through Fuller's earth. The changes in viscosity and in carbon residues of residuum brought about by the above methods of treatment indicate clearly that certain heavy constituents of the crude oils, not affected by the acid treatment, are removed by filtering through Fuller's earth. It is pointed out that this fact may be of some practical significance in the refining of Mid-Continent crudes for the production of the better grades of cylinder stock.

16. LOW SPEED HIGH-PRESSURE FRICTION TESTS WITH A KINGSBURY MACHINE.—W. F. Faragher and Ronald Reamer, Mellon Institute, Pittsburgh.

A discussion is given of data on frictional coefficients, obtained with a Kingsbury oil-testing machine, in which speed was maintained at 7·4 feet per minute, and pressure was varied over the range of 2000–4000 lb. per sq. in. A number of mineral oils and several compounded lubricants were studied.

17. A GRAPHICAL STUDY OF JOURNAL LUBRICATION.—H. A. S. Howarth, Kingsbury Machine Works, Philadelphia, Pa.

This study is based on W. J. Harrison's paper, "The Hydrodynamic Theory of Lubrication." The oil pressures and friction are represented quantitatively by simple diagrams, showing the effects of varying the speed, oil viscosity, eccentricity and clearance. A few examples are given, showing the practical application of this study to the case of a journal surrounded by the bearing.

18. THE MECHANISM OF PARTIAL LUBRICATION.—A Review by R. E. Wilson, Mass. Inst. of Tech., Cambridge.

This subject is presented from the standpoint of the author's recently published papers on the subject, and some additional material is brought forward.

19. DISCUSSION BY.—W. H. Herschel, Bureau of Standards, Washington, D.C.

THE ORIGIN OF PETROLEUM.

20. ORIGIN, MIGRATION AND ACCUMULATION OF OIL AND GAS.—R. E. Somers, University of Pittsburgh, Pittsburgh, Pa.

The inorganic theories of origin, such as that of Mendeléef, are geologically impossible for large amounts of oil and gas. The organic theories better account for the commercial pools. Effects of geological alteration continue beyond the first formation of oil and gas, and according to White's laws, lighter fractions are added with increasing pressure and longer time. The geologically older oils are lighter in weight, and the younger oils are heavier.

Oil passes from the source bed shales into the reservoirs because of compacting of the shales, and perhaps because of a differential capillarity.

Accumulation into pools starts when the reservoirs are loose and uncemented, and is then due principally to differences in specific gravity between oil, gas, and water. It continues as the reservoirs become cemented, but gravitation is either assisted by other influences, such as gas pressure, or else replaced by capillarity.

21. THE PROBABLE MOTHER SUBSTANCE OF PETROLEUM.—Reinhardt Thiessen, Bureau of Mines, Pittsburgh, Pa.

Many scientists interested in the origin of petroleum hold that the bituminous rocks are the mother substances, so if it is assumed that these are the source of petroleum, a better knowledge thereof is of practical value. All bituminous rocks may be divided into two great classes; namely, coals and bituminous shales.

Coals.—Coal is composed of a residue of the various plant compounds, of which the cellulose and lignocelluloses contribute the larger and most important part. With these are always associated the residues of the more resistant plant components, such as resin, waxes, fats, and oils—but in a minor part.

Bituminous Shales.—Under this heading are grouped the cannel coals, boghead coals, cannel slates, kerosene shales, torbanite, and oil shales. These may be classified definitely according to the origin of their predominant components. Five classes have been recognised, namely humic shales, spore shales, coorongite shales, Kiskiminetas shales, and Green River shales.

Humic Shales.—Cellulose degradation matter, or humic matter, forms the predominant component, with an admixture of resinous, spore and cuticular matter as minor constituents of the humic shales.

Spore Shales.—The predominant component of the organic matter of the spore shales is composed of spore exines of a variety of plants. Arbitrarily, they may be classified into spore-cannel coals, spore-cannel slates, and spore-oil shales, according to the proportion of mineral matter present in them. To this group belong the ordinary cannel coals, cannel slates, and oil shales, which constitute the bulk of the shales.

Coorongite Shales.—These shales comprise torbanite, kerosene shales, and bogheads. These have long been described as being formed by algæ. It has now been found that they were formed by an organism similar or like that living to-day in certain salt lakes and salt lagoons, and give rise to deposits known as coorongite. The organisms have certain characteristics of the blue-green algæ, but their position is still under investigation, and is not definitely known.

Kiskiminetas Shales.—This class is best represented by a cannel coal found near Kiskiminetas Junction, also in the Pottsville formation. The origin of the predominant component is unknown.

Green River Shales.—These are found in tertiary and grataceous formations, their main organic component is ceric. Those of the Green River formation of Utah and Colorado are representative of the group.

22. DISCUSSION BY.—C. F. Mabery, Case School of Applied Science, Cleveland, Ohio, and W. H. Emig, University of Pittsburgh.

RESEARCH ON PETROLEUM.—Report of the Committee on Research. W. F. Faragher, Mellon Institute, Pittsburgh, Pa. Discussion by Van H. Manning, American Petroleum Institute, New York, N.Y.

Abstracts.

Compiled by H. Moore, M.Sc., F. G. P. Remfry, B.A., D.Sc., A.I.C., and T. A. Smith, B.Sc., F.I.C.

General.

The Surface Tension of Petroleum.—C. K. Francis and H. T. Bennett. *J. Ind. Eng. Chem.* 1922, **14**, 626.

The surface tension of various fractions of petroleum oil is found to increase with increase of specific gravity, boiling point and viscosity. With rise of temperature, however, there is a decrease of surface tension approximately of 0·05 dynes per cm. for each °F. The presence of the small quantities of fatty acids and amorphous and crystalline wax usual in petroleum oils does not appear to affect the surface tension. In the case of various lubricating oils of enormously different viscosities the differences in surface tension, as determined against air, was practically negligible, and was therefore no measure of their values as lubricants.

F. G. P. R.

A Possible Origin of Oil.—C. C. Rae. Trans. Am. Inst. Mining. *Met. Eng.* 1922, No. 1137–P. 4 pp.

Humus acids and associated organic compounds are considered to be the intermediate step between petroleum oils and complex plant compounds, and as evidence in support of this theory is quoted the observation of Binney in 1912, that petroleum oil was formed by decomposition of peat mosses at Down Holland in England in the presence of infiltered sea water. Haseman and others have also noted the association of swampy areas near the sea, with asphalt and similar hydrocarbons. There appears to be some relationship between the silica and humus acids content of river waters, probably in some way connected with their colloidal nature, but on admixture with sea water organic compounds are deposited owing to coagulation by electrolytes. It would also appear that silica is deposited at the same time, judging from the presence of finely divided silica in Californian shales associated with petroleum. The humus acid carried by rivers every year totals millions of tons, and the gradual transformation of this in petroleum hydrocarbons probably occurs under several different conditions, in which pressure, temperature, alkalinity, salts and the presence of catalytic agents all vary. In the western parts of the United States are found oil shales containing organic compounds intermediate between humus acids and petroleum, which are convertible into the latter by means of heat.

F. G. P. R.

The Oil Supply of the United States.—*Bull. Am. Assoc. Pet. Geologists* 1922, **6**, 42–6.

The paper is a report of a committee, composed of members of the American Association of Petroleum Geologists and the U.S. Geological Survey, on the oil reserves in the present producing regions as well as in probable and possible areas of future production The estimates arrived at bring the total to nine billion barrels of oil, recoverable by the methods at present in use, dating from Jan. 1, 1922.

F. G. P. R.

ANALYTICAL DISTILLATION OF PETROLEUM AND ITS PRODUCTS.—E. W. Dean, H. H. Hill, N. A. C. Smith, and W. A. Jacobs. *Bureau of Mines Bull.* 1922, **207**, 82 pp.

Detailed descriptions are given of the methods employed at the U.S. Bureau of Mines for distilling crude petroleum and its products, determining specific gravities, viscosities, etc. A description is given of the cloud test, tests for water and methods of dehydration. The original paper should be consulted for details. F. G. P. R.

DETECTION OF BENZOL IN BENZINE.—P. Schwarz. *Chem. Ztg.* 1922, **46**, 410.

The test is qualitative and gives a positive reaction with as little as 5% of benzol in the benzine; 5 c.c of the latter are shaken with a mixture of 1 c.c. of aniline, and 1 c.c. of 94–96% (vol.) ethyl alcohol. The presence of more than 5% of benzol brings about complete solubility. It is advantageous to use the fraction boiling 80–110° C. in place of the benzine itself. F. G. P. R.

THE MUTUAL SOLUBILITY OF HYDROCARBONS, FATS AND LIQUID SULPHUR DIOXIDE.—E. Zerner, H. Weisz, and H. Opalski. *Z. angew. Chem.* 1922, **35**, 253–256.

Hard glass tubes were sealed at one end and drawn out at the other, and weighed quantities of the oils under investigation and of liquid sulphur dioxide introduced, after which they were sealed up while in a freezing mixture. They were then immersed in a bath, by which their temperature could be controlled, and the point of precipitation observed. The commercial sulphur dioxide used contained 98·85% of sulphur dioxide, 0·56% of water and 0·67% of oily residue. Toluol, *n*-tetradecane, *n*-pentatriacontane, decahydronaphthalene, tetrahydronaphthalene and American kerosene were tested, as also the fatty oils, castor, rape, olive, linseed, and bone fat. The graphs in all cases show a maximum. Paraffins are the least soluble, naphthenes more so, and aromatic and unsaturated compounds are easily soluble. The solubility decreases with increase in molecular weight. Sulphur dioxide shows great solubility in hydrocarbons. H. M.

TEMPERATURES OF COMBUSTION.—J. Bronn. *Z. angew. Chem.* 1922, **35**, 328.

Temperatures of combustion may be calculated with approximate accuracy from the heat of combustion of gases and the specific heat of the products of combustion. But the temperature of combustion of hydrogen in pure oxygen is given in the Chemiker-Kalender as 6670° C., and from this it would appear that the specific heat of steam is taken at 0·485, which is correct at the temperature of 800° C. This figure for the temperature 3000° C. is, however, 0·713, which would give a temperature of combustion of 4500° C., and the value for 4000° C. may be taken at 0·85, which would give the temperature of combustion as 3900° C. The temperatures for hydrogen, carbon monoxide (5000° C.), methane (4400° C.), and acetylene (6200° C.) are calculated in this manner. The use of compressed methane for welding is of advantage, as the gas does not give such a high temperature of combustion as to be injurious to the metals operated on, while at the same time supplying a large quantity of heat. The velocity of flame propagation of gases has great influence on their temperatures of combustion, except in so far as it may be affected by flameless combustion. The velocities of flame propagation of ethylene, acetylene and benzene are shown to be different. H. M.

ON THE POSSIBILITY OF ISOLATING BITUMEN IN ITS ORIGINAL FORM FROM BITUMINOUS ROCKS.—E. Hentze. *Z. angew. Chem.* June 23, 1922, pp. 330–331.

Instead of the process of Narbutt (*J. Soc. Chem. Ind.*, June 30, 1922 p. 452A), which consisted in the extraction of the bitumen from the rock, the procedure consisted in dissolving the rock from the bitumen. Green kieselguhr from Luneburger Heide and Esthonian oil shale, or "Kukkersite" were tested, the respective bitumen contents being about 4% and 28%. About two kilos. of each sample were repeatedly treated in the cold with dilute hydrochloric acid, and washed with warm water, for a period of fourteen days, to free them from calcium and iron. The kieselguhr was then treated for four weeks, and the "Kukkersite" for fourteen days with dilute, and then with 40% hydrofluoric acid to remove silica and clay. The residua were found not to be free from ash, and were extracted with chloroform to obtain the bitumen free from inorganic matter. To compare the extracted bitumen with that present in the rock before isolation, the extracts were mixed intimately with clay and calcium carbonate to represent the composition of the original rock, and distilled in a glass retort, as also the original rocks. The isolated bitumen from kieselguhr gave 1% less, and the "Kukkersite" 5% less volatile products than the original bituminous rock, but the light-boiling products in the resultant tar were much greater in the case of the isolated bitumens, and the cokes produced by the latter were richer in carbon. A process of polymerisation seems to have occurred in the extracted bitumens, followed by cracking on distillation. It is proposed to attempt the isolation of the bitumens in an unchanged state by electro-osmotic separation from a colloidal suspension. H. M.

THE PROBABILITY OF REACTION BETWEEN SULPHUR AND PARAFFIN WAXES IN OIL-BEARING STRATA.—M. A. Rakusin. *Petroleum*, 1922, **18**, 581–582.

4·1725 gr. of paraffin wax, of m.p. 56° C. was heated in a glass retort with 0·2810 gr. sulphur to 190°–205° C. for 18 hours. The escaping vapours containing hydrogen sulphide passed through two U-tubes. The first was filled with pumice moistened with ammonia. In this tube ammonium sulphide and polysulphides were formed. The latter were decomposed with hydrochloric acid, and the separated sulphur collected. The second U-tube contained calcium chloride. It was found that the amount of sulphur which entered into reaction was less than 50% of the sulphur expended. From this it appeared that in oilfields which produce both oil free from paraffin wax, and oil containing paraffin wax, the oil free from wax would have a smaller sulphur content than the oil containing wax. This was found to be the case in the Roumanian and Grozny oilfields, the respective percentages of sulphur in the oil being, for Roumanian oil, 0·042% and 0·112%, and for Grozny oil, 0·212% and 0·782%. For the de-paraffination of crude petroleum oil a temperature of 200° C. is necessary, which will only be reached at corresponding depths. In the higher and cooler strata the paraffin wax is not attacked by sulphur, and remains in the oil, or may crystallise out. H. M.

EVAPORATION LOSS OF PETROLEUM IN THE MID-CONTINENT FIELD.—J. H. Wiggins. *Bull. Bureau Mines*, No. 200.

Extensive investigations were made on the oil fields as well as in the laboratory to determine the loss on different types of plant by evaporation under various conditions. The loss is found to be very considerable, as, for example, a loss of 2·32% for nine days occurred in a lease storage

tank, while a total loss of 8% is possible. The principal occasions of loss by evaporation are the filling of tanks by overshot connection, in which case spray is formed and may be carried away by the wind; the filling of tanks by bottom connections, which causes ebullition and a consequent accelerated evaporation of the oil; the storage of oil in small tanks, protected or unprotected from sun and the free circulation of air, and the storage of oil in large standard steel tanks of a capacity up to 55,000 barrels. There are also to be considered the losses occasioned in dehydrating tanks, and in all subsequent stages of handling. In the field tests the loss was found by measurements of the tanks on consecutive dates, and the figures obtained were checked by tests of the specific gravity of the oil, and by distillation tests, giving the quantities of the fractions. In laboratory tests it was found that the rate of evaporation of the first 10% gasoline fraction from a crude averages 1·91 on the rate of evaporation of the gasoline from the undistilled crude. The rate of evaporation of pure gasoline and a mixture of 10% of gasoline blended with 90% of a heavy lubricating oil of 22° Beaumé was in the proportion 2.57–1. In cases in which the 10% gasoline was blended with heavier gasoline or mineral seal oil the pure gasoline evaporated little faster than when it was blended. Californian gasolines of the same gravity evaporate approximately twice as fast as those from the other fields. The evaporation of petroleum is generally not due so much to "wild gas" as to the light gasoline fractions. The bulletin gives a large number of illustrations and graphs and also formulæ for the calculation of evaporation.

H. M.

PREVENTION OF EVAPORATION OF VOLATILE LIQUIDS.—F. A. Howard, C. I. Robinson, and J. M. Jennings. U.S.P. 1,415,351–2. May 9, 1922. Also *Nat. Petr. News.* 1922, **14**, 28, 43.

Loss by evaporation of crude petroleum or refined petrol in storage tanks is prevented by covering the surface of the oil with a layer, about 1 in. deep, of a permanent foam, consisting chiefly of corn-starch, glycerine and gelatine. The ingredients are beaten up together at a temperature of 90° F., and at the same time air is blown into the mass, whereby a froth containing 50% of air is obtained, weighing 4¾ lbs to the gallon (American). The seal has been found to be permanent for at least twelve months, but, being soluble in water, must be protected from rain. A special device is attached to the storage tanks, whereby water takes the place of oil as it is removed, and thus obviates disturbance of the layer of foam.

F. G. P. R.

GASOLINE FROM NATURAL GAS IN 1921 IN U.S.—*Anon., Chem. Met. Eng.* **26**, 1110.

The output of natural-gas gasoline in 1921 increased 23% over that in 1920. Seventy-five per cent. of the output was recovered at compression plants and the remainder at absorption plants. Although the output was greater the amount of gas treated was 5% less, indicating increased efficiency.

T. A. S.

A P.I. SCALE FOR GRAVITY OF LIQUID PETROLEUM PRODUCTS.—*Chem. Met. Eng.* **26**, 1120.

The attention of the American Institute having been drawn to the confusion arising from the use of two "degrees Beaumé," the one having a modulus of 141·5, and the other having a modulus 140, it has been agreed that the former should be adopted by the American Petroleum Industry, and known as the A.P.I. Scale. Gravities will be expressed on this scale in "degrees A.P.I."

T. A. S.

MODERN METHODS OF CONCENTRATING SULPHURIC ACID.—P. S. GILCHRIST. *Chem. and Met. Eng.* 1922, **26**, No. 25, 1159–62.

Four systems of concentrating sulphuric acid are described: (1) cascade, (2) tower, (3) combination surface evaporation and tower concentration, (4) hot air submergence type. For the concentration of sludge acid from the refining of petroleum oils the last system is the only really practical one. Owing to contamination with carbonaceous matter concentration must be carried out at the lowest possible temperature, in order to avoid decomposition of the sulphuric acid and liberation of SO_2. The underlying principle of the process is concentration by blowing hot air, at a temperature of about 1200° F., through the acid. The operation is carried out in two stages, in the first the concentration is not carried beyond 50 to 55° Bé. in order to avoid frothing, whilst in the second an acid of 66° Bé. is produced. The acid sludge is delivered into the top of a 30 feet high tower packed with staggered brickwork, and falls into a concentration pan at the base. Hot air at 1200° F. is blown into the acid in the pan and passes up through the tower, carrying with it hydrocarbon vapours. The concentrated acid is continuously run off from the pan, cooled and pumped to the top of a similar tower for the second stage concentration, the resulting finished acid coming out of the pan at about 110° F. below its boiling point, and with a gravity of 66° Bé. The hot air for the process is obtained by blowing air through a combustion chamber in which oil is burned. In concentrating acid from 35° Bé to 66° Bé, starting from cold, about 35 gals. of oil are consumed per ton of finished acid.

F. G. P. R.

DRYING OILS FROM PETROLEUM.—H. A. Gardner and E. Bielouss. *J. Ind. Eng. Chem.* 1922, **14**, 619.

The authors found that by chlorinating certain fractions of petroleum oil and then dechlorinating the product, an oil was obtained which had drying properties equal to those of raw linseed oil. On exposure to weather, however, the durability of the petroleum product was not entirely equal to that of linseed oil. The best results were obtained with a fraction of petroleum known as "non-viscous neutral oil," and in this case chlorination proceeded so smoothly "at the temperature employed" that no catalyst was required. Dechlorination was brought about by simply heating to 250° C. without the assistance of any catalyst or HCl absorbing agent.

F. G. P. R.

FUEL OIL VISCOMETERS. W. H. Herschel. *Chem. and Met. Eng.* 1922, **26**, No. 25, 1175–7.

Owing to the difficulty of obtaining Redwood Admiralty viscometers in the United States, and the unsuitability of the Saybolt Universal viscometer for very high viscosities the Saybolt Furol type was developed in 1920. The paper gives an account of the standardisation and calibration of the latter as well as the calibration of the Redwood instrument. For mathematical details the original paper should be consulted, but the conclusions arrived at are that the most probable equations are—kinematic viscosity $= 0{\cdot}0220t - \frac{2{\cdot}03}{t}$ (Saybolt Furol) and $0{\cdot}0239t - \frac{0{\cdot}403}{t}$ (Redwood) where t is the time of flow in secs., and kinematic viscosity is expressed in c.g.s. units. These equations and that for the Saybolt Universal viscometer—kinematic viscosity $= 0{\cdot}00220t - \frac{1{\cdot}80}{t}$—have been used in calculating conversion tables for the three instruments.

F. G. P. R.

ETHEREAL SULPHATES, ALCOHOLS AND ESTERS.—S. B. Hunt. E.P. 173,538. June 30, 1920.

Gas containing unsaturated hydrocarbons chiefly of the olefine type, as well as saturated hydrocarbons, such as that produced by cracking petroleum oils, is treated with sulphuric acid of 1·84 sp. gr., with or without admixture of a neutral liquid having a solvent action on the gas such as a hydrocarbon oil insoluble in the acid, at temperatures varying from 10° to 40° F. For the production of alcohols, mainly secondary, the alkyl sulphates formed are diluted with water and distilled. Esters are formed by adding acetic acid, calcium acetate, etc., diluting and distilling. F. G. P. R.

LOW BOILING CHLORINATED HYDROCARBONS, MANUFACTURE OF.—H. O. Traun. E.P. 156,139 of Dec. 31, 1920.

By passing the vapour of high-boiling hydrocarbons, such as gas oil, mixed with hydrochloric acid gas or chlorine through a tube heated to 600–1200° C. pyrogenic decomposition of the hydrocarbon and addition of the halogen takes place simultaneously. Low-boiling chlorine compounds of hydrocarbons result which are useful as solvents, or may be converted to diolefines by splitting off hydrochloric acid. F. G. P. R.

ALDEHYDE ACIDS, ALDEHYDES, HYDROCARBONS AND SOAPS.—C. P. Byrnes. E.P. 173,750 of Dec. 21, 1920.

Oil vapour, such as that from crude petroleum, gas oil, shale oil, or that produced by cracking heavy oils is mixed with air and passed through a catalytic material under regulated temperature conditions. Diluents such as steam may be added to the reaction mixture. After distilling off the higher portions for motor spirit the residue is treated with alkali hydroxide to remove aldehyde acids. The remainder, consisting of alcohols, aldehydes and waxes, is suitable as a lubricant. F. G. P. R.

LIQUID HYDROCARBONS; PROCESS OF MANUFACTURING FROM FISH OIL.—Kyuhei Kobayashi. E.P. 170,264 of July 24, 1922. (Application 11,712-21. Convention Oct. 13, 1922.)

Fish oils, *e.g.*, herring, salmon or sardine oils are decomposed by heating with Japanese acid clay to a temperature not lower than 500° C. Glycerine and fatty acids are first formed, and are subsequently broken down, forming liquid hydrocarbons from which light oil, illuminating oil and other fractions may be separated by distillation. The operation is carried out by mixing the fish oil (400 lb.) with acid clay (400 lb.) and covering the mixture in a cast iron retort with a further 400 lb. of clay. The mixture is then distilled. The distillate is a green fluorescent oil, resembling natural petroleum. It contains small quantities of naphthenic and fatty acids. On fractionation the distillate yields 30% boiling to 150° C., 50% boiling from 150–275 C., and 20% neutral residue. The Japanese acid clay used is closely related to fuller's earth, and its composition may be expressed by the formula $Al_2O_3.6SiO_2.xH_2O$, in which x is not less than 6. It acts as a catalyser. T. A. S.

MANUFACTURE OF DIOLEFINES AND POLYMERS THEREOF.—H. O. Traun. E.P. 156,116 and 156,122 of Dec. 30, 1920.

156,116. Equimolecular quantities of gases of the ethylene and acetylene series are heated to 350–400° C. under a pressure of 2–10 atmospheres, and combine to form diolefine compounds. By raising the pressure and increasing the time of the reaction a polymerisation of the

diolefines to rubber-like substances is brought about. Inert gases or vapours, such as benzene, may be introduced so that a pressure of 10–50 atmospheres may be employed, and contact agents such as oxides or hydroxides of the alkali or alkaline earth metals or oxychlorides may be introduced to assist the reaction.

156,122. Halogen derivatives of hydrocarbons, which are capable of yielding diolefine compounds by a simple splitting off of hydrochloric or hydrobromic acid, are formed by passing a mixture of the hydrocarbon vapour and the halogen or its hydride through a tube heated to a high temperature such as 500–800° C. The presence of catalysts such as silicon alloys, silicates or platinum accelerate the reaction. F. G. P. R.

Oils—Process of Treating Hydrocarbon.—H. T. Maitland. Assr. to Sun Co., Philadelphia. U.S.P. 1,425,882–3. (Application April 15, 1919.)

1,425,882. A water-insoluble organic acid capable of being saponified is made from petroleum oil by subjecting a heavy distillate, such as lubricating oil stock, to treatment by sulphuric acid and separating off the acid sludge. The remaining acid containing oil is treated with alkali, and the latter run off and acidified with a relatively weak acid, whereby the alkali soaps are decomposed and insoluble organic acids liberated. 1,425,883. The acid sludge is masticated with a continuous supply of fresh hot water, until the free acid content of the sludge is sufficiently reduced to leave the water insoluble saponifiable oil above specified. F. G. P. R.

Stable Foam for Preventing the Evaporation of Stored Liquids.—J. M. Jennings, Assignor to Standard Oil Company. U.S.P. 1,423,719–21 of July 25, 1922. (Appl. Nov. 22, 1920, May 24, 1921, and Oct. 26, 1921.)

The foams described in the patents are: (1) An aqueous solution containing 25% glue, 10% glycerine, 2% ferrous sulphate, and a minute proportion of preservative; (2) A permanent foam having, as its continuous phase, a liquid consisting of less than 15% water, glycerine, and a body-forming material; (3) A liquid containing 40–81% glucose, 6–12% water, 5–10% glue, and a small proportion of a setting agent. T. A. S.

Mineral Oil and Carbohydrogen Gas Extracting Process.—G. Schneiders. U.S.P. 1,418,098 of May 30, 1922. (Appl. Sept 2, 1919.)

A passage driven into bituminous and gaseous deposits is protected by means of an air-dam, which prevents the leakage of gas and oil into the passage. The gas and oil are removed from the deposit by means arranged in the passage. The air-dam is moved along into the deposit as gas and oil are removed. T. A. S.

Mineral Oil and Carbohydrogen Gas Extracting Apparatus.—G. Schneiders. U.S.P. 1,418,098 of May 30, 1922. (Appl. Aug. 15, 1921.)

Oil-bearing sand is removed from its deposit, and simultaneously subjected to separating treatment. The process consists in dislodging the sand by a jet of fluid, so that the action of the jet and friction of the grains removes the adherent bitumen. T. A. S.

Gasoline—Apparatus for Recovery of Gasoline from Casing—Head Gas.—W. R. McGinnis. U.S.P. 1,418,876 of June 6, 1922. (Appl. Aug. 23, 1919.)

Two scrubbers each provided with baffles, refrigeration apparatus, and a sprinkling head, are used. Means are provided for passing the

gas down one scrubber and up the other, and means for reversing the said gas movement. T. A. S.

OLEFINES—PROCESS OF OXIDISING, ETC.—C. Ellis. U.S.P. 1,418,368 of June 6, 1922. (Appl. May 17, 1918.)

Ketones and fatty acids are obtained from the acid extract of olefines from cracked petroleum, by diluting the acid extract, and treating it with dilute nitric acid. T. A. S.

WASTE SULPHURIC ACID—PROCESS FOR TREATMENT OF.—S. Hilpert, Assr. to Himself and Deutsch-Luxemburgische Bergwerke Und Hutten Aktiengesellschaft. U.S.P. 1,427,386 of Aug. 29, 1922 (Appl. July 9, 1921.)

The waste sulphuric acid obtained in the purification of light oils is treated with an aromatic hydrocarbon to recover resinous products. T. A. S.

SULPHUR COMPOUNDS; PROCESS OF OBTAINING FROM MINERAL OILS.—J. C. Clancy, Assignor to the Nitrogen Corporation. U.S.P. 1,423,711 of July 25, 1922. (Appl. July 14, 1920.)

The sulphur compounds, separated from the oil as ammonia-sulphur compounds through the agency of liquid ammonia, are recovered. T. A. S.

SLUDGE ACIDS; METHOD OF TREATING.—I. Hechenbleikner and T. C. Oliver. U.S.P. 1,423,766–7 of July 25, 1922. (Appl. Jan. 25, 1922.)

The method consists in heating a weak sludge acid with agitation, thereby concentrating to an intermediate strength and further concentration without agitation. T. A. S.

OIL—MINERAL . . . DERIVATIVES.—H. T. Maitland, Assr. to Sun Co., Philadelphia. U.S.P. 1,425,884–5. (Appl. April 15, 1919.)

The water-insoluble saponifiable organic acids described in U.S.P. 1,425,882–3 are claimed as new compounds, one being liquid and the other solid at ordinary temperatures. They are insoluble in water, practically odourless, do not become rancid, and contain less than one per cent. of sulphur. They are readily saponifiable to substances soluble in water in all proportions, and in mineral oils totally and partially respectively, and as such have the characteristics of soaps derived from animal and vegetable oils. F. G. P. R.

RECOVERY OF SULPHURIC ACID FROM ACID SLUDGE.—V. C. C. van Voorhout. Holland P. 2217 of Nov. 1, 1917.

The acid sludge is diluted with water and heated in lead-lined autoclaves for at least two hours, under a pressure of ten atmospheres, by which means the sulphuric acid is caused to separate from the tar. The requisite pressure is obtained by introducing carbon dioxide into the autoclave under high pressure and heating by means of a steam jacket. F. G. P. R.

CRACKING.

GASOLINE—PRODUCTION OF SYNTHETIC.—R. Cross. *Oil and Gas J.* **20**, Nos. 41, 74, 76, 78, 80 (1922).

There are three principal sources of gasoline, about 70% being obtained from natural crude petroleum, 23% by cracking processes, and 5% from

natural gas. Cracking is not a simple decomposition of the hydrocarbon molecule, lighter and higher boiling constituents being both formed during cracking processes. Cracking is therefore accompanied by polymerisation. The gases produced in cracking vary with the method used. Gasoline made by $AlCl_3$ process has the lowest percentage of olefines. Gasoline made by the pressure distillate method is much superior to gasoline made by the high temperature vapour-phase processes.

T. A. S.

PRODUCTION OF GASOLINE BY CRACKING HEAVIER OILS.—E. W. Dean and W. A. Jacobs. Technical Paper 258. U.S. Bureau of Mines.

Experiments have been made on the effects of varying the conditions during the cracking process. A furnace was used similar in principle to that of Whitaker and Rittman (*cf. J. Soc. Chem. Ind.* 1914, **33**, 682), but with several improvements introduced. The apparatus consists of a drip feed lubricator, delivering oil into the upper end of a vertical electrically heated tube, to the lower end of which is attached condensing and receiving apparatus. The tube is packed with a chain to obtain contact and dispersion. A thermocouple pyrometer measures the temperature of the tube wall halfway down the tube. The temperature is regulated by a rheostat which controls the current passing through the heating element. Experiments were made using Pennsylvanian distillate of a gas oil type, Mid-Continental residuum fuel oil, Pennsylvanian kerosene distillate from which fractions boiling below 175° C. had been removed by topping, and a kerosene distillate from which products boiling below 200° C. had been removed by topping. Increase in the rate of feed produced a decrease in the degree of cracking, when the furnace wall temperatures were kept constant. The evidence supports the theory that cracking occurs mainly at the contact surface from which heat is supplied to the oil. Other conditions being maintained constant, it was found that the yield of cracked products decreased as the temperature increased, the loss varying from 6% to 40% in the experiments. The conditions which would produce commercial yields of gasoline are accompanied by losses of over 10%. The sp. gr. of the cracked product is influenced by the temperature, the gravity falling until the temperature reaches approximately 558° C., at which temperature the gravity of the product is at a minimum. Increase in temperature produces increased gravity of the product, and if the temperature is sufficiently high the sp. gr. of the product will be greater than that of the raw material. The percentage of gasoline formed increases with increased temperature. The advantage of high temperatures is, however, compensated by the greater loss during the operation. The sp. gr. of the recovered gasoline fraction is raised with increase of temperature, probably on account of the formation of aromatic hydrocarbons. The figures indicate that at pressures of 50 lbs. and over, the degree of unsaturation of the gasoline fraction tends to decrease as the temperature increases. Presumably this is due to the formation of aromatic compounds. The ratio of gasoline formation to recovery loss is tabulated, as this point is of great importance in commercial working. When pressure is varied, other conditions being maintained constant, it is found that the percentage of cracked oil recovered generally tends to decrease as the pressure under which cracking takes place increases. Using a temperature of 570° C. the sp. gr. reaches a minimum value, when the pressure is 50 lbs. per sq. inch. The percentage of gasoline formed generally increases with increase of pressure. The sp. gr. of the gasoline fraction tends generally to increase as pressure increases, presumably owing to formation of aromatic hydro-

carbons. The degree of unsaturation of the gasoline fractions tends to decrease as pressure increases. The ratio of gasoline formation to recovery loss is tabulated. The pressure favourable for minimum ratios varies between the limits 50 and 150 lbs., depending upon the temperature. The effect of variation in the rate of feed, other conditions being constant, is as follows. Increase in the rate of feed, which is equivalent to shortening the time during which reaction is allowed to occur, increases the percentage of cracked oil recovered. The general tendency is for the sp. gr. of the recovered oil to decrease as the rate of feed increases. The effect of change of the rate of feed upon the percentage of gasoline formed is irregular. Increase in the rate of feed tends to decrease the sp. gr. of the gasoline fraction when the degree of cracking is considerable. The degree of unsaturation of the gasoline tends to increase as the rate of feed is increased. The effect of the ratios of gasoline recovery to recovery loss indicate that gasoline can be produced more economically by maintaining high furnace temperatures and a high rate of feed than by using lower temperatures and rates. When a residual oil is used as raw material a much larger amount of carbon is formed during the process than when a distillate is employed. The difference in behaviour between Mid-Continental residual and paraffin base distillate is shown in a series of graphs. Comparing the behaviour of kerosene with that of gas oil as a raw material for the cracking process, the percentage recovery is greater with the kerosene, whilst the gasoline yield is less. The gasoline from the kerosene fraction is lower in sp. gr. than that prepared from gas oil, whilst the degree of unsaturation of the gasoline fraction is less on the products obtained from kerosene, the general conclusion being that the two products are about equally suitable, the gas oil being slightly easier to transform but not quite as economical as to losses in recovery. Comparisons were also made of the cracking of Pennsylvanian and Naphthene base oils, the results indicating that the Naphthene base oils require a higher temperature than the paraffin base oil. Experiments indicate that the common practice of cracking oil containing naphtha fractions is not economical. A more efficient procedure would be to separate the naphtha in a topping still, passing the residue to a cracking plant, and adding the naphtha to the gasoline obtained from the cracking plant. By this method a greater yield of gasoline is obtained than when the naphtha containing distillate is passed through the cracking plant. It is found that the residuum obtained after the removal of gasoline from a cracked product yields less favourable results both as regards cracked oil recovery and percentage of gasoline formed than the results obtainable from fresh oil. A larger percentage of carbon is deposited on re-cracking than in the first process. It is observed that the volume of gas formed is closely related to the loss in recovery of cracked oil. H. M.

PREVENTION OF CARBON DEPOSITS ON METAL CATALYSTS.—The Chemical Fuel Co. of America. E.P. 160,466 of March 3, 1921.

In catalytic reactions where gaseous or vaporous carbon compounds are passed over heated metal catalysts, such as nickel, iron, cobalt, etc., the surface of these is kept bright and free from carbon deposit by means of causing electric sparks to bombard the catalytic material. In order to do this a metal electrode is placed at a suitable distance from the catalyst, which acts as the other electrode, and an electric current of 10,000–20,000 volts and 100,000 frequency employed. F. G. P. R.

HYDROGENISING HYDROCARBONS.—A. J. Stephens. E.P. 174,106 of Aug. 14, 1920.

Vapours from unsaturated hydrocarbon oil, produced by distillation,

are mixed with hydrogen either within or outside the still and condensed to a liquid in the presence of a catalyst, such as nickel, by a pressure of 4 to 5 atmospheres. In some cases the metal walls of the condensing apparatus may have sufficient catalytic action. Saturated hydrocarbons similar to commercial petroleum products are obtained. F. G. P. R.

PETROLEUM OR OTHER HYDROCARBON OILS—PROCESS AND APPARATUS FOR TREATING.—J. P. Persch. Eng. Pat. 181,034. (Appl. 34,419-20.)

The oil is treated with a hot gaseous fluid, preferably air, the apparatus being arranged so that an injector effect is obtained, the oil being drawn from a bulk, passed through tubes mixed with the air, and returned to the bulk. In an example given oil of 38° Bé. was treated for 30 mins. with compressed air at 390° F. The temperature of the oil did not exceed 152° F. After treatment 95% of the oil distilled over, the distillate yielding 60% gasoline, 7% lubricating oil, and 3% heavy lubricating oil. The gasoline yield from the original oil was 25%. An oil of 11–15° Bé., after 30 minutes' treatment, was much less viscous and retained this property indefinitely. T. A. S.

PETROLEUM OILS—IMPROVEMENTS IN, OR RELATING TO, APPARATUS FOR THE DISTILLATION AND CRACKING OF PETROLEUM OILS.—S. L. Gartland and A. E. Gooderham. Eng. Pat. 179,645 of Feb. 8, 1921. (Appl. 4479-21.)

Mixed steam and petroleum oil is fed into a retort, the vapours being drawn off by a compressor and condensed under pressure, the pressure under which the condensation takes place being varied according to the final product desired. The retort is heated by a step-by-step process, the liquid volatile at each temperature being drawn off at that temperature, and the pressure in the retort being maintained at about that of the atmosphere. As the temperature of distillation rises, steam, superheated to that temperature is admitted to the retort. It is stated that 75% of the petroleum oil can be converted into low-boiling naphtha. T. A. S.

HYDROCARBON OILS—AN IMPROVED PROCESS FOR DISTILLING AND CRACKING.—S. L. Gartlan and A. E. Gooderham. Eng. Pat. 179,644 of Feb. 8, 1921. (Appl. 4496-21.)

The hydrocarbon oil is heated in a retort, the vapours being withdrawn so that the distillation takes place at approximately atmospheric pressure, and without appreciable change in the hydrocarbon molecules. Steam is admitted, the steam being superheated to the temperature of the retort as the boiling point of the contents of the retort rises. The condensation of the vapours takes place under pressure, the pressure being raised in accordance with the rise in temperature of distillation. The retort is heated by a step-by-step process and maintained at each temperature until all the contents volatile at that temperature have been removed. It is claimed that from Mexican crude petroleum 75% of the matter treated can be recovered as naphtha, averaging about 72° Bé. T. A. S.

HYDROCARBONS—IMPROVEMENTS RELATING TO ART OF CRACKING.—The Kansas City Gasoline Company. Eng. Pat. 160,161 of Feb. 22, 1921. (Appl. 6014 of 1921. Int. Conv. March 12, 1920.)

High-boiling hydrocarbons are mixed with a relatively small quantity of carboniferous material—*e.g.*, bituminous coal and heated with agitation in a still or retort. Arrangements are made for passing forward light distillate, and the return of heavy distillate for further cracking. Fresh

high-boiling hydrocarbon is added to the carboniferous material to replace that distilled off. The distillation is carried on at atmospheric pressure out of contact with the air. In an example given the cracking temperature was 600–1000° F. T. A. S.

DISTILLATION OF OILS—IMPROVEMENTS RELATING TO.—Trent Process Corporation. E.P. 171,075 of July 13, 1922. (Appl. April 13, 1921. Conv. Nov. 4, 1920.)

Oil is mixed with finely-divided fuel of relatively high volatile content, *e.g.*, coal, anthracite bituminous coal or lignite, and distilled in a retort at a temperature approximating to 350° C. It is claimed that considerable cracking takes place, increasing the gasoline and light oil content of the oil. Any carbon residue is obtained as a dry dust. During distillation the mixture is agitated by rabbles. The mixture consists of 1 part oil, 2 parts coal by weight. T. A. S.

CRACKING HYDROCARBON OILS.—Gulf Refining Co. E.P. 174,085–6–7–8.

When cracking oils by distilling under pressure, in any form of still, gas, such as that formed during cracking, natural or casing-head gas, or steam, is injected into the oil in such a way as to cause it to circulate over the heated surface and thus prevent or remove deposits of carbon, which action may be assisted by the addition of sand, gravel, metal filings, etc. F. G. P. R.

CRACKING HYDROCARBON OILS.—D. T. Day. U.S.P. 1,422,038 of July 4, 1922.

Heavy oils are vaporised in the usual way, and the vapours led into a compressor maintained at such a temperature that on compression none of the vapours are condensed to liquid form. The degree of compression applied is such as to raise the temperature of the vapours above that at which cracking occurs. Moreover, heating takes place uniformly throughout the mass, as is well known in compressing gases, thereby avoiding the local overheating that must result when applying heat externally to tubes or boilers and the danger consequent thereon. From the compressor the vapours are led to an insulated chamber, where they are maintained at the same temperature and pressure until complete conversion has taken place and then led to condensers in the usual way. F. G. P. R.

HYDROCARBONS—PROCESS FOR PRODUCING LIGHT.—C. R. Burke. Assr. to L. P. Burke. U.S.P. 1,426,149 of Aug. 15, 1922. (Appl. Jan. 16, 1912.)

Heavy oil is distilled under atmospheric pressure, and the vapours led into a compressor, where they are subjected to a pressure sufficient to raise their temperature to that at which cracking takes place. By varying the pressure the degree of cracking can be regulated and a resulting product of any desired specific gravity and character can be obtained at will. F. G. P. R.

ALUMINIUM CHLORIDE—SEPARATING . . . FROM HEAVY HYDROCARBONS.—C. O. Hoover. Assr. to the Hoover Co., Chicago. U.S.P. 1,426,081 of Aug. 15, 1922. (Appl. April 3, 1922.)

In order to recover aluminium chloride from its combination with heavy hydrocarbon oils, such as occurs when being employed as a catalytic agent in cracking, the combination product is dissolved in a suitable solvent and the mixture thus produced subjected to sublimation. F. G. P. R.

PETROLEUM—DISTILLING . . . . UNDER HIGH PRESSURE.—C. E. STOCKFORD. Assr. to Standard Oil Co., New York. U.S.P. 1,425,712 of Aug. 15, 1922. (Appl. May 8, 1915.)

The fire chamber and flue beneath a horizontal high-pressure still is so arranged that the whole of the length of the still bottom is exposed to direct flame and fire gases. The distance between the bottom of the still and the top of the furnace bridge wall should be upwards of one-third of the diameter of the still. F. G. P. R.

HYDROCARBONS—PROCESS FOR PRODUCTION OF LOW BOILING.—J. C. Black. U.S.P. 1,426,813 of Aug. 22, 1922. (Appl. Dec. 12, 1919.)

Oil is heated in a tube still to cracking temperature, but under a pressure of not less than 35 atms.; whereby vaporisation is prevented. The heated oil is then passed into a larger chamber, kept completely filled with oil and under the same pressure as the still, wherein carbon is deposited and thence passes to an atmospheric pressure still, when the light portions vaporise and are led to condensers. The heavier unvaporised portions are returned, together with fresh oil, to the cracking still. F. G. P. R.

CRACKING HYDROCARBONS.—O. M. Alexander. U.S.P. 1,407,619 of Feb. 21) 1922.

The vapours from crude petroleum or high-boiling distillates are passed through a zone heated to 500°, at about atmospheric pressure, at a rate sufficiently rapid to prevent the decomposition of a large part of the vapours. The vapours are fractionally condensed and unchanged products of high-boiling point are returned for retreatment. T. A. S.

GASOLINE—PROCESS FOR PRODUCING.—J. C. Clancy. Assr. to the Nitrogen Corporation. U.S.P. 1,423,709 of July 25, 1922. (Appl. July 31, 1919.)

The vapour of hydrocarbon oil is cracked by submitting it to the catalytic action of mercury vapour. T. A. S.

CATALYZING OILS—PROCESS AND APPARATUS FOR, AND REVIVIFYING THE CATALYST.—E. V. Owen. Assr. to The Hoover Co. U.S.P. 1,427,626 of Aug. 29, 1922. (Appl. March 31, 1922.)

Low-boiling hydrocarbons are produced by passing high-boiling hydrocarbons with aluminium chloride hydrocarbon catalyst through a catalyzing chamber maintained at a suitable temperature. After passing through the catalyzing chamber the oil is separated from the catalyst and returned to the chamber. The returning oil passes along the outer wall of the catalyzing chamber and is heated to maintain the chamber at the correct temperature. Low-boiling hydrocarbon vapours are removed from the chamber. T. A. S.

CRACKING OIL—PROCESS OF AND APPARATUS FOR—G. L. Hoxie. U.S.P. 1,418,713 of June 6, 1922. (Appl. Aug. 9, 1918.)

The process consists in heating a small portion of the oil up to cracking temperature, and immediately mixing the same with a large volume of oil at a substantially lower temperature, the low-boiling products being distilled off the large volume of oil. Several forms of apparatus are proposed for doing this. T. A. S.

PETROLEUM—APPARATUS FOR TREATING.—L. Clark. U.S.P. 1,418,621 of June 6, 1922. (Appl. March 14, 1918.)

Oil is fractionated or cracked by heating in the presence of a heated fluid, *e.g.*, flue-gases. The heated gases circulate the oil and entrain the vapour. The apparatus consists of a shell suspended in a chamber to provide an annular passage way. The bottom of the shell is an open cone. A central pipe conducts the heated vapour down the shell and circulates the oil through the annular space. The apparatus is arranged so that any temperatures and pressures may be used. T. A. S.

HYDROCARBON MATERIAL—PROCESS OF MAKING UNSATURATED.—A. A. Wells. U.S.P. 1,418,414 of June 6, 1922. (Appl. Oct. 9, 1916.)

Hydrocarbon oils are cracked under high pressure until still bottoms of a tarry consistency are formed. These still bottoms are distilled, and the distillate recracked, under a pressure lower by at least one atmosphere than the original cracking pressure. By this process hydrocarbon material is obtained rich in unsaturated hydrocarbons, which can be extracted with sulphuric acid without undue rise in temperature.
T. A. S.

PETROLEUM VAPOUR—APPARATUS FOR TREATING.—D. W. Hoge. U.S.P. 1,418,375 of June 6, 1922. (Appl. June 14, 1917.)

Petroleum vapour is converted into gasoline by passing it through a continuous conduit, built up of straight sections and heated in a bath of molten metal. The conduit system is bodily removable from the bath.
T. A. S.

HYDROCARBONS—METHOD OF TREATING.—W. F. Rittman. U.S.P. 1,419,125 of June 6, 1922. (Appl. Aug. 3, 1917.)

Oil is fed in at the bottom of a vertical retort, where it is vaporised. The vapours pass upwards through the retort and are cracked. The maintenance of a large body of oil at a high temperature is avoided as is selective distillation. The primary object of the invention is the cracking of oils boiling below 600° F. T. A. S.

LOW-BOILING HYDROCARBONS—PROCESS FOR THE CONTINUOUS PRODUCTION OF—FROM PETROLEUM OILS.—R. W. Hanna. U.S.P. 1,419,378 of June 13, 1922. (Appl. Jan. 19, 1920.)

The oil is first distilled under reduced pressure, so as to prevent cracking, the heavy oils being preferably continuously withdrawn. The high-boiling distillates to be cracked are carried forward to an apparatus kept at a pressure higher than atmospheric and a temperature sufficient to crack the same. The addition of a solvent oil to the oil to be cracked prevents the formation of deposits in the system. The cracked products and residues are continuously removed. T. A. S.

OILS AND PROCESS FOR CRACKING.—R. D. George. U.S.P. 1,420,832 of June 27, 1922. (Appl. Sept. 14, 1921.)

The process consists in heating to cracking temperature heavy hydrocarbon oils in continuous flow through an upright still. The heated oil is delivered from the top of the still to a separating chamber wherein is maintained a pool of hot oil above the level of the still. In the separator the volatile portions are removed and condensed. The residual oil is freed from carbon in a carbon separator and returned to the bottom of the still. The amount of free carbon in the cleaned oil is kept below 1%.
T. A. S.

CRACKING TAR AND PETROLEUM OILS TO BENZOL AND BENZINE.—Naamlooze Venootschap Hollandsche Benzol en Benzine Maatschappij. Holl. P. 5162 of July 15, 1920.

Hydrocarbon vapours mixed with steam are passed through carbonaceous material, such as coke, maintained at temperatures varying from 300° to 1200° C. in a vertical retort. The latter is arranged so that a zone of low temperature (300° C.) surrounds one of high temperature (500–1200° C.). F. G. P. R.

MOTOR FUELS.

HYDROGENATION OF ACETYLENE FOR THE PREPARATION OF FUEL OILS.—Keiji Oda. *J. Chem. Ind. Japan* 1921, **24**, 1161–6.

A mixture of equal volumes of acetylene and hydrogen is passed over a catalyst, maintained at 25–35° C., composed of 10 parts by weight of $Ni(NO_3)_2$ and 1 part of $Hg(NO_3)_2$ mixed and heated to 250–300° C., in order to convert to oxides, and finally reduced by heating in the presence of hydrogen for eight hours at 300° C. 52% of the theoretical yield of oil was obtained, of which a little more than two-thirds was light oil and the rest heavy oil, boiling at 200–300° C. F. G. P. R.

ALCOHOL FUELS FOR INTERNAL-COMBUSTION ENGINES.—W. Petchell. *S. African J. Sci.* 1921, **18**, 143–6.

The values of alcohol, alcohol with acetylene dissolved in it, ether, benzol, etc., as motor fuels are discussed. "Natalite" and Shell spirit were tested on a 1.5 kw. engine at full, two-thirds, half, and one-third load. In percentage efficiency "Natalite" was 75·6, 79·6, 80·3 and 86·8, compared to Shell at these loads, in calorific value 72% and in a practical mileage test approximately 80%. With a stationary engine a compression of 200 lbs. per sq. in. could be employed with ether as fuel, and by using a hit-and-miss governor this would be more economical than petrol and would approximate to a Diesel engine in thermal efficiency. F. G. P. R.

DETONATION CHARACTERISTICS OF SEVERAL BLENDED MOTOR FUELS.—T. Midgley, J. & T. A. Boyd. *Nat. Petr. News* 1922, **14**, No. 27, 59–68.

Owing to the continually expanding practice of adding aromatic hydrocarbons, such as benzol, toluol and xylol, as well as alcohol, to commercial motor fuels for the purpose of reducing the tendency to detonate, a study has been made of the detonating characteristics of blends of alcohol and alcohol-benzol mixtures with paraffin hydrocarbons. The bouncing pin method (*J. Soc. Automotive Eng.*, Jan., 1922, p. 7) was employed for measuring the intensity of the detonations, and kerosene was used in place of petrol owing to its greater detonating characteristic. When blended with aromatic hydrocarbons kerosene and petrol agree sufficiently closely with one another in their detonation curves to make the use of the former permissible in these experiments. For the purpose of comparison a standard fuel, composed of xylidine dissolved in kerosene, was employed in place of a standard engine, since it is found that a given fuel will give widely different results in different engines, but comparable results in any one engine. One per cent. of xylidine is equal to 15% of benzol in eliminating detonation, and therefore very small amounts can be used. Absolute ethyl alcohol was used in the tests in order to be able to make the alcohol-kerosene blend. Details are given of the method of carrying out the tests, and tables and curves are appended showing the

results obtained. Less than 20% by volume of benzol, added to a commercial petrol, has very little effect in obtaining smoother working of the engine, but above this amount the effect increases rapidly. Contrary to Ricardo's statement (*Autom. Eng.* (*London*), March, 1921, p. 96) toluol is found to be more effective than benzol, and xylol than toluol, on the basis of volume. On the same basis alcohol is considerably more effective than benzol, 35% of the former being equal to 55% of the latter when blended with kerosene. If, however, molecular quantities are employed in place of volumes, alcohol does not show such a superiority, the proportion now being 35 to 42. It was expected that the detonation curve of a mixture of equal quantities (volume or molecular) of benzol and alcohol with kerosene would fall midway between those of each separately with kerosene, but experiments conclusively proved that the mixture of the three was invariably less efficient than the calculated mean of the two binary mixtures. F. G. P. R.

PREPARATION OF MOTOR FUELS CONTAINING ALCOHOL.—The Chemical Fuel Co. of America. E.P. 159,880 of March 3, 1921.

In order to remove water from commercial alcohol and so render it miscible with low-boiling hydrocarbons and benzol, a small amount of fusel oil—0·25 to 1·0% of the total mixture—is added to the ingredients, and the whole subjected to distillation. The first 40% of distillate contains hydrocarbons, alcohol and the majority of the water, the next 30% hydrocarbons, alcohol and nearly all the rest of the water, whilst the last 30% consists of hydrocarbons, alcohol and a mere trace of water. The last portion is used for motor fuel and the first two are returned for reuse in the process, after rectification of the alcohol to its original strength. F. G. P. R.

MOTOR FUEL.—F. Hostettler. E.P. 174,360 of Jan. 20, 1922.

By the addition of an oxygen carrier, such as wood spirit, a fuel for internal-combustion engines may be made from raw oil distillates, such as earth or rock oils and tar oils from pit-coal, brown-coal, lignite, peat, etc. As an example 40–50% of tar distillate boiling between 75–130° C., 40–50% of raw oil distillate boiling between 50–360° C. and 3–5% of dehydrated wood spirit are mixed, distilled together and filtered. F. G. P. R.

MOTOR FUEL—HYDROGENATING HYDROCARBONS.—M. Melamid. E.P. 174,321 of Nov. 9, 1921, and 180,625 of May 25, 1922. (Additional to E.P. 171,367 and 161,390.)

174,321. Low-boiling hydrocarbons are produced and hydrogenated by passing the vapour of heavy hydrocarbons mixed with a large excess of hydrogen over metallic tin situated between baffle plates heated to redness. Atmospheric or only very slight pressures may be employed.

180,625. Similar products are obtained by using metals or alloys, which melt at the reaction temperature, such as soldering metals or bismuth alloys melting below 700° C. Pressures above atmospheric are employed. F. G. P. R.

ALCOHOL FUEL.—IMPROVED.—J. Hawes. E.P. 184,607 of Aug. 24, 1922. (Appl. 14,781 of 1921.)

The fuel consists of ethyl alcohol (96–98%), 96 parts; petroleum naphtha, 2 parts; nitrobenzene, 1 part; and amyl alcohol, 1 part. T. A. S.

FUEL FOR INTERNAL-COMBUSTION ENGINES—IMPROVED.—Aktiengesellschaft für Anilin Fabrikation. E.P. 169,428 of June 17, 1921. (Appl. 16,742 of 1921. Int. Conv. Sept. 20, 1920.)

The fuel consists of a mixture of hydronaphthalenes with suitable cyclohexanes. A good fuel consists of 50–70% methylcyclohexane and 50–30% tetrahydronaphthalene. T. A. S.

APPARATUS CONNECTED WITH AN INTERNAL-COMBUSTION OR OIL ENGINE FOR CONVERTING CRUDE OIL INTO FUEL; IMPROVEMENTS RELATING TO.—T. D. Key. E.P. 183,600 of Aug. 2, 1922. (Appl. 12,491 of 1921.)

The removal of impurities from crude oil is facilitated by heating it by means of the exhaust gases from the engine. The apparatus is so arranged that a thermo-siphon is set up. The oil passes through filters to remove suspended matter, and sumps are provided for the removal of water and asphalt. T. A. S.

FUEL FOR USE IN INTERNAL-COMBUSTION ENGINES; IMPROVEMENTS RELATING TO.—H. R. Ricardo. E.P. 183,577 of July 26, 1922. (Appl. 11,953 of 1921.

The introduction of water into the cylinder of an internal-combustion engine, with the object of reducing the temperature at which the engine works, cooling the piston, inlet and exhaust valves and other advantages, is attained by using benzol-acetone water mixtures with or without the addition of ethyl or methyl alcohol. At least 8% of water is required. A suitable fuel consists of 60% ethyl alcohol, 20% acetone, 10% benzole and 10% water, the latter being in solution. T. A. S.

FUEL—MOTOR.—F. W. Rohrs. U.S.P. 1,425,135 of Aug. 8, 1922.

A liquid fuel for internal-combustion motors is composed of 88% of a light petroleum distillate, 9% of benzol, and 3% of acetone to act as an oxygen carrier. F. G. P. R.

FUEL—LIQUID.—B. R. Tunison. Assr. to U.S. Industrial Alcohol Company. U.S.P. 1,423,048–50. (Appl. April 12, 1920.)

The fuels consist of (1) A liquid fuel denser than kerosene, consisting of a relatively heavy aliphatic hydrocarbon and alcohol; (2) a liquid fuel consisting of a petroleum distillate heavier than kerosene, an ester of an organic acid and a mono-hydric alcohol, the heavy oil forming three-quarters of the mixture; (3) a liquid fuel consisting of a petroleum distillate heavier than kerosene and an aromatic alcohol. T. A. S.

FUEL FOR USE IN INTERNAL-COMBUSTION ENGINES.—M. C. Whitaker. Assr. to U.S. Industrial Alcohol Company. U.S.P. 1,423,058. (Appl. Nov. 12, 1919.)

The fuel consists of benzol 17–35 parts, gasoline 20–50 parts, ethyl acetate 5 to 20 parts, and alcohol more than 20 parts by volume. T. A. S.

DISTILLATION AND REFINING.

ON THE DISTILLATION OF A MIXTURE OF BENZENE, TOLUENE AND METAXYLENE.—L. Gay. *Chim. et Ind.*, May, 1922, pp. 851–854.

This mixture corresponds approximately to the "light oils" of coal tar distillation, the metaxylene forming the greater part of what is known

commercially as solvent naphtha. The distillation takes place in two successive dephlegmating columns, and the process may be so arranged as to separate practically pure metaxylene at the base of the first column, while the mixed toluene and benzene are separated in the second column, or to extract practically pure benzene from the top of the first column, leaving a liquid mixture of toluene and metaxylene to be separated by further distillation. By a series of triangular graphs the exchange of heat units is explained, showing under what circumstances the first, and when the second mode of operation is preferable. H. M.

THE LIMITS OF THE ADSORPTIVE RECEPTIVITY OF FLORIDIN.—M. A. Rakusin. *Petroleum*, 1923, **19**, 797–798.

Experiments were made to determine the minimum quantity of Floridin (aluminium-magnesium hydrosilicate) necessary to transform a dark-coloured crude oil solution into a polarimetrically clear solution by the infusorial method. A 1% solution of Grozny petroleum free from paraffin was discoloured by standing with 5% of Floridin for 24 hours. A further addition of Floridin did not improve the result. By lengthening the time of treatment the same result was obtained by 1% of Floridin (in 96 hours). It is suggested that much smaller quantities of Floridin would have the same action if the time of contact were prolonged, and that its action resembles catalytic action, or that of the trypsines in digestion, in which the quantity of enzyme multiplied by the time of action is a constant. H. M.

PETROLEUM REFINING EQUIPMENT—CORROSION OF.—R. R. Mathews and P. A. Crosby. *Chem. Met. Eng.* **26**, 1119–20.

Corrosion is stated to be mainly due to formation of free HCl by hydrolysis of $MgCl_2$. Most of the hydrochloric acid is liberated at temperatures ranging from 175–250° C. Corrosion is most serious in those stills in which live steam is used. Remedial measures are discussed, including addition of ammonia to vapour systems and the more difficult complete dehydration of crude oil before distillation. T. A. S.

THE SULPHURIC ACID TREATMENT OF PETROLEUM PRODUCTS.—C. J. von Bibra. *Chem. and Met. Eng.*, 1922, **27**, No. 2, 66–72.

The author deals with the purely chemical problems involved in refining petroleum products with sulphuric acid, and does not touch on the practical application of this method. The paper is divided into two main portions, "General Theory" and "Factors affecting acid treatment." In the former section the constituents of petroleum are described in turn, and the reactions which take place when each is subjected to treatment with sulphuric acid. The subject matter is subdivided in the following paragraphs—"The sulphuric treatment outlined"—"Paraffins and naphthenes"—"Bituminous substances"—"Organic acids"—"Sulphur bodies"—"Nitrogen bodies." In the second half of the paper the factors which affect the treatment are discussed, and consist essentially in the variations in the constitution of oils, and in the quantity, strength, purity and method of applying the acid, as well as in the effect of temperature, time and light on the reaction.

F. G. P. R.

METHODS OF RECOVERING GASOLINE FROM UNCONDENSED STILL VAPOURS.—D. B. Dow. *Nat. Petr. News*, 1922, **14**, No. 22, 28–31.

Uncondensed vapours are classed as "sweet" or "sour gas," according as they are formed during the distillation of the light ends of a crude oil,

or by cracking of heavier portions in tar or pressure stills. In skimming plants practically all uncondensed vapours are formed during the distillation of the first 15% of the crude, and as much as 30 gallons of gasoline per 1000 cu. ft. of vapour has been found, *i.e.*, 90% of the vapours were condensible. On an average, however, 7 gallons per 1000 cu. ft. was obtained in practice on a works' scale.

Cracked vapours contain a large amount of fixed gas and yield an average of below 3 gallons of condensible gasoline per 1000 cu. ft. They also contain sulphur which must be removed before condensation. Atmospheric temperature, the nature of the oil being distilled, the temperature of the condensate at the worm-end and the vacuum carried on the run-down lines, are the chief factors that determine the gasoline content of the uncondensed vapours.

In summer the production of vapour is greater than in winter, owing to the temperature of air and cooling water being higher. A high vacuum also increases production of vapour, and not more than two inches of water should be employed.

Experiments with Cushing crude oil showed that at worm-end condensate temperatures from 75° F. to 120° F. the recoverable gasoline content of the uncondensed vapours per barrel of crude distilled varied from 0·05 gallons to 1·01 gallons when using 2 inches (water) of vacuum, 2 sq. ft. of condenser surface per gallon distilled per hour and absorbing in "mineral seal" oil.

The straight condensates produced at each particular worm-end temperature were redistilled in a steam still, and it was found that the quantity of uncondensed gasoline produced decreased with each successive rise in worm-end temperatures, until at 120° F. practically all was found to have been already taken off at the crude still. Between 50° F. and 90° F. worm-end temperatures the following formula gives the number of gallons of gasoline that can be condensed from the vapours from every barrel of crude oil distilled.

$$\frac{A}{1000} \times \left(F - \frac{T}{2} \times 0{\cdot}03\right)$$

When A = gallons of gasoline per 1000 cu. ft. of gas. F = cu. ft. of gas formed at 70° F. worm-end temperature. T = difference in °F. between the worm-end temperature and 70° F. Various oils in cracking and pressure stills gave from 80 to 200 cu. ft. of gas per barrel distilled, containing from 3 to 7 gallons of condensible gasoline per 1000 cu. ft.

Tables are given showing the gasoline recovered and methods employed at 10 representative refineries, the recovery ranging from 0·05 to 0·84 gallons per barrel of crude and amounting to a total of 128,651 gallons per day from 355,486 barrels of crude distilled. For removing the vapours positive-pressure, rotary blowers or exhausters give the best results. The gases are removed at a point between the condenser and the tail box through a riser attached to the gas main, a liquid seal being provided to prevent air being sucked back from the tail box. A small pipe dipping two inches below water should be attached to the riser to prevent the building up of pressure in the still or vacuum in the gas line. Traps are installed on the latter prior to the exhausters, where any condensate is removed, and the gases washed free from sulphur by water. At the present time 50,000,000 gallons of gasoline from an estimated possible 170,000,000 gallons are recovered in U.S.A.

PART II.—*Nat. Petr. News*, **14**, No. 23, 87–93.

The usual methods for recovery of liquefiable products consist in (*a*) compression and cooling of the vapours, (*b*) passing the vapours through heavier liquid hydrocarbons, or (*c*) a process in which the uncondensed vapours after treatment by compression are washed with heavy oil. The compression process is the one most widely used, most plants compressing in one stage at below 100 lbs. per sq. in., and never exceeding 800 lbs., and depending on a naphtha absorption system for removing gasoline from the residual gas. In small plants treating only the "sweet" vapours (*i.e.*, those with very low sulphur content) the intake of the compressor will produce the desired vacuum on the run-down lines, but where a sulphur removing tower is required it is necessary to employ an exhauster as well. Sour gas is often led to boilers and burned without attempting to remove gasoline. Where two-stage compression is employed the power expended in the low-stage compression should equal that expended in the high stage, thus obtaining an equal discharge temperature from each compressor, provided the intake temperatures of the gas is the same in each case. There is thus an equal amount of cooling to be performed after each stage, necessitating equal condenser areas and temperature of cooling water. In practice about 0·8 sq. ft. of condenser surface are provided per 1000 cu. ft. of gas treated daily, but as gas is very seldom metered on refineries it is often better to calculate from the horse-power used in compression. The figure should be about four sq. ft. per horse-power. The gas, under compression, is passed through water-cooled condensers, and the condensate collected in "accumulator" tanks under the same pressure. Blending the condensate with naphtha is performed in the "accumulator" by pumping the required amount of naphtha in from below, or spraying from above, the latter giving better results owing to absorbent action on the uncondensed supernatant vapours. The best results, however, are obtained by pumping naphtha into the top of the cooling coils, and is known as "hot blending." The blended product is finally mixed with straight-run gasoline. Blending naphtha generally has a final boiling point about 437° F., and a gravity of 50–52° Bé., whilst the blended product may have 400° F. and 58–60° Bé. For use in the absorption process the same grade of naphtha may be used, or a gas oil of about 35° Bé., from which the dissolved gasoline is subsequently removed by steam distillation. In a plant treating about one million cubic feet of gas per day with naphtha of 50° Bé., five absorption towers, 10 × 30 feet are employed. The gas is forced in under low pressure at the bottom of the first tower and discharges at the top, flowing counter-current to the naphtha, and passing through the whole series of towers. The naphtha is either circulated continuously in each tower until sufficiently saturated, and then replaced by fresh absorbent, or passed from tower to tower through the whole series in counter-current to the gas. Since the absorbing power of naphtha or gas oil decreases rapidly above 85° F. the temperature of both gas and absorbent should be well below this before coming in contact with each other, since a certain amount of heat is generated on absorption taking place. Very low temperatures are useless, because it is impossible to hold a large part of the absorbed vapours when brought up to normal temperatures, and this excessive evaporation causes actual loss of heavier material. In constructing absorbing towers the diameter should be in such proportion to the volume of gas to be treated that the velocity of the latter does not exceed 75 feet per minute through the towers. The height is regulated by the degree of saturation of gasoline in the absorbent oil required. Wooden or earthenware baffles are

necessary where the sulphur content of the vapours is higher than that found in natural gas. When using gas oil as the absorbent the saturated material is freed from gasoline by means of live steam and returned to the absorbing towers continuously, or is sent to pressure stills and the whole cracked to gasoline. By this means, however, there is probably a loss of absorbed gasoline due to cracking to permanent gas. In the natural gas industry gas oil is preferable to naphtha as an absorbent, owing to the large amount of fixed gas to be treated, but on refineries where still vapours containing relatively small amounts of fixed gas are handled, the use of naphtha presents many advantages.

PART III.—*Nat. Petr. News*, 1922, **14**, No. 24, 53–7.

Crude oils which contain large amounts of sulphur, such as Mexican, Gulf Coast and some Californian oils, make it necessary to wash the still vapours before these are stripped of gasoline. Where pressure stills are employed this is especially necessary owing to decomposition of heavy sulphur compounds into vaporous ones by heat and pressure. The amount of sulphur in still gases has been found to vary from 600 to 3000 grains per 100 cu. ft. of gas in refineries distilling varying admixtures of Mexican Crude. The two methods principally in use for removing sulphur and its compounds, are (1) washing the gases with water and caustic soda solutions; and (2) passing the gases through iron oxide suspended in water. In the former scrubbing towers, 20 × 4 feet, are employed and a caustic soda solution of 15° Bé. (1·115 sp. gr.). 600 lbs. of sodium hydroxide made up to this strength will treat on an average 1½ million cu. ft. of vapour a day for three to four weeks, and reduce the sulphur content from 3000 to 60 grains per 100 cu. ft. The efficiency of the solution goes off rather quickly but can be revived by blowing with air. In the iron oxide treatment the oxide is held in suspension in the water by agitation with air, which also serves in reviving the suspension by converting iron sulphides to oxides. The advisability of adding a recovery plant to an already existing refinery will depend on the efficiency of the condensers in use, on the temperature of the cooling water, and the nature of the crude distilled. Tests should be carried out on the uncondensed vapours either by compression, absorption in oil, or in activated charcoal, and the results of these will indicate whether the additional plant should prove commercially sound.

F. G. P. R.

HYDROCARBONS—IMPROVEMENTS IN REFINING LIQUID . . .—A. E. Dunstan. E.P. 184,281 of May 12, 1921. (Appl. 13,515-21.)

In order to remove sulphur compounds from liquid hydrocarbon oils the latter are subjected to a preliminary treatment with liquid alkali, then agitated with an alkaline hypochlorite solution, such as sodium hypochlorite, containing free alkali, and finally treated again with liquid alkali. The preliminary alkaline treatment removes some sulphur compounds and acidic compounds, which would decompose the hypochlorite, whilst the final treatment eliminates the majority of the sulphur compounds oxidised by the hypochlorite, and not removed by the latter.

F. G. P. R.

FRACTIONAL DISTILLATION OF CRUDE OIL; IMPROVEMENTS RELATING TO.—P. Mather. E.P. 182,827–8. (Appl. 1785 of June, 1921.)

Crude oil, before delivery to a continuous still, is pre-heated, both by hot residue and still vapours. The oil is sprayed or dripped on to coils containing the residues or vapours, and provision is made for the partial

evaporation of the crude oil in the heat-exchangers, and the admission of steam or otherwise. A series of heat exchangers are provided to ensure the economical use of the heat, and the quantity of hot residue passing through the heat exchangers is controlled by a valve on the hot residue main and a valve admitting hot residue to the heat exchanger, both operated by a worm. The apparatus operates in conjunction with a vertical still so arranged that vapours may be drawn off at different levels, and consequently at different temperatures. In 182,828 the construction of the heat-exchanger is more specifically described.

T. A. S.

SEPARATING OILS; IMPROVEMENTS IN THE PROCESS OF.—Trent Process Corporation. Eng. Pat. 167,738. (Appl. 10,828-21.)

The process is designed to deal with such emulsions as base sediment refining acids and sludges. When such emulsions are agitated with comminated carbon the oil and carbon unite, forming an agglomerate from which the water and the other substances separate. The carbon used is fine enough to pass through a 100–200 mesh sieve, and the quantity used is 21 lbs. of carbon to 1 lb. of oil, in the mixture. The oil-carbon mixture sinks in still water, and may be used direct as fuel, or distilled to recover the oil. T. A. S.

HYDROCARBONS—IMPROVEMENTS IN AND RELATING TO PROCESSES FOR REFINING.—J. Demant. Eng. Pat. 179,610 of Feb. 4, 1921. (Appl. 4136-21.)

The acid treatment of hydrocarbons for the removal of impurities is varied, so that instead of the foreign matters being lost in solution in concentrated acid they are polymerised and recovered as high-boiling oils from the hydrocarbons by subsequent distillation. To do this the hydrocarbon is treated with more dilute acid at suitable temperatures. The action is catalytic, the acid is unchanged, and may be used repeatedly. By treating 90% benzole with 10% of its weight of sulphuric acid of 46° Bé. at about 65–70° C. for 5–6 hours, and subsequent treatment with soda and distillation, the pure benzole is recovered, and on distilling the residue with steam, oil and valuable varnish are obtained. In the case of coal tar naphtha treatment with acid of 46–48° Bé at 110° C. for 5–6 hours converts the indene and coumarone into a viscous oil of high value, having a freezing point of (–10°) to (–15°) C. T. A. S.

DISTILLATION OF LIQUIDS, SUCH, FOR EXAMPLE, AS TAR AND OILS—IMPROVEMENTS IN.—J. L. Wilson. Eng. Pat. 180,347 of Nov. 17, 1920. (Appl. 32,391-20.)

The oil is distilled in a continuous still, inert gas being passed through the distilling liquid. Separation of the distillates is effected by fractional condensation. Heat is conserved by using the condensers and still residue coolers as preheaters for the incoming oil. The arrangement of the still head prevents the return of any condensate to the still. Vapours from the preheaters and those not condensed by the tar cooled condensers, are passed through water-cooled condensers. T. A. S.

EMULSIONS; PROCESS FOR RESOLVING.—The Sharples Specialty Company. Eng. Pat. 180,447 of June 1, 1922. (Appl. 6959-21.)

Emulsions are resolved by adding to them colloidal solutions soluble in the continuous phase, and which tend to reverse the emulsion. Soap can be added to a water-in-oil emulsion by preparing a reagent soluble in oil, and yet containing soap. Such a reagent containing 25% sodium

soap, 10% water, and 65% oleic acid, may be prepared by adding excess of oleic acid to a solution of caustic soda of 30° Bé. The addition of 1% of a reagent containing 40% water, 35% rosin, and 25% rosin soap, with stirring to a viscous oil emulsion, resulted, on centrifuging, in a separation of the water from the emulsion. T. A. S.

PETROLEUM OILS; IMPROVED STILL FOR FRACTIONALLY DISTILLING.—E. C. R. Marks. E.P. 182,247 of June 6, 1922. (Appl. 11,438-21.)

The still is particularly designed for the refractionation of gasoline-kerosene cuts. The fraction is heated by means of hot residues, from which it was originally distilled. A long horizontal still is divided by vertical partitions, and heated by tubes through which the residuum flows. The gasoline-kerosene fractions flow along this still, and steam is admitted into each portion. Each section is provided with its own vapour exit. T. A. S.

DESULPHURISING OILS; AN IMPROVED MEANS.—A. C. Nesfield. E.P. 183,527 of July 24, 1922. (Appl. 9157-21.)

The oil is treated in a thin film with chlorine gas. The treatment takes place in a tower, down which the oil descends being caused to form films by flowing over cones. The chlorine is circulated, but has to be replaced in time, owing to loss of efficiency. The sulphur is precipitated in flocculent form, and is separated from the oil by filtration, centrifugal treatment or removed by means of water. The reaction is expedited by the addition of quicklime, and by preheating the oil to a suitable temperature—for certain oils 100–120° C. The precipitate consists of sulphur compounds in addition to sulphur, and may contain bituminous matter. T. A. S.

HEAT INTERCHANGE—MEANS OF EFFECTING . . . BETWEEN TWO FLUIDS, PARTICULARLY APPLICABLE FOR USE IN DISTILLING OILS.—T. E. Robertson. (Communicated by Power Specialty Co., of New York.) E.P. 179,493 of Nov. 26, 1920. (Appl. 235-22.)

In apparatus for heating fluids, such as pipe stills or steam superheaters, the pipes nearer the furnace receive more heat than those more remote with consequent danger of over-heating the contained fluid. In order to equalise the amount of heat transmitted to the fluid along the whole system of pipes the latter are enclosed in corrugated casings, on which the depth of the corrugations vary more or less inversely, whilst the thickness of the body of the casing varies directly as the temperature difference between the heating and heated fluids. F. G. P. R.

LIQUIDS OF DIFFERENT DENSITY (*e.g.*, OIL AND WATER)—APPARATUS FOR SEPARATION OF.—W. H. Bateman. E.P. 179,209 of Nov. 8, 1920. (Appl. 31,575-20.)

A "tail-box" for the separation of oil and steam condensates produced during distillation of oils in the presence of steam is so constructed that it may be attached to any form of separating chamber, and is divided vertically into four compartments: (1) Water discharge; (2) and (3), oil discharge; and (4) sight box with a glass front, showing respective levels of the liquids in the separating chamber. Compartment (1) is connected with the bottom of the separating chamber, and has an overflow pipe of adjustable level, so that the column of water therein is balanced by the column of oil and water in the separating chamber. Compartment (2) is connected to the separator just below the top level of the oil therein, and to compartment (3) on an exact level therewith,

thus providing a liquid seal in the connection between (2) and the separator to prevent gas loss. Compartment (3) is furnished with an oil-discharge pipe to storage. The sight box (4) has connections in its upper part with the oil compartment (2), and in its lower part with the water compartment (1). F. G. P. R.

DISTILLING OILS—PROCESS AND APPARATUS FOR.—J. T. Fenton. U.S.P. 1,424,998 of Aug. 8, 1922.

For the purpose of topping oils the still is constructed with a bottom in the form of descending steps down which the oil is made to flow in a thin stream. A weir at the top of the steps, having a vertically movable gate over which the oil flows, forms a preheating reservoir, whilst at the bottom is situated a well, in which the depth and temperature of the oil are made to regulate valves on the oil inflow pipes leading into the preheating reservoir. F. G. P. R.

FRACTIONATING APPARATUS.—H. L. Allan. U.S.P. 1,421,886 of July 4, 1922.

A series of vessels, connected at their upper parts by tall vertical vapour pipes acting as air condensers, are arranged in the form of a cascade. Each vessel is divided horizontally into three chambers, the lower and upper of which are connected by a series of pipes projecting almost to the top of the latter, and every vessel is connected to the next lower one in the cascade by a reflux pipe leading from the base of the upper chamber to the lower chamber of the adjacent vessel. Steam may be introduced into the central chamber for heating purposes, and the lower chamber is provided with an open steam coil to assist distillation. Vapours from the still enter the lowest vessel in the cascade, and pass upwards through the connecting air condenser pipes from vessel to vessel. Condensate collects in the upper chamber of each vessel, flows by way of the reflux pipe to the lower chamber of the next lower vessel, and thence through the series of pipes into the upper chamber thereof, and so on back to the still. By this means condensate from the upper vessels come in contact with the hotter vapours and condensates in the lower vessels, and are thereby partly or wholly re-evaporated, thus producing better fractionation. Condensate may be withdrawn from the lower chamber of each vessel and passed to storage. F. G. P. R.

FRACTIONAL CONDENSATION—PROCESS OF.—J. F. W. Schulze. U.S.P. 1,418,885 of June 6, 1922. (Appl. June 21, 1919.)

A series of superimposed dephlegmator columns are arranged so that the vapour from each column heats the condensate from the column above it without coming in actual contact with the liquid. The condensates from each section are run off separately through overflow pipes. T. S. A.

DESULPHURISING OILS—METHOD OF.—J. C. Clancy, Assignor to The Nitrogen Corporation. U.S.P. 1,423,712 of July 25, 1922. (Appl. July 17, 1920.)

The method consists in heating the oil with ammonia vapour and steam. T. A. S.

DESULPHURISING OIL—PROCESS OF.—J. C. Clancy, Assignor to the Nitrogen Corporation. U.S.P. 1,423,710 of July 25, 1922. (Appl. July 8, 1920.)

The oil is treated with liquid anhydrous ammonia to cause the formation of an ammonia-sulphur compound, which is then separated. A catalyst may be used to cause the sulphur to combine with the ammonia. T. A. S.

OIL—REFINING.—P. T. Sharples. U.S.P. 1,416,891 of May 23, 1922. (Appl. Aug. 24, 1921.)

The residue obtained by the removal of the lower boiling constituents from crude petroleum without substantial cracking is treated to obtain the precipitation of the wax whilst maintaining the fluidity of the oil so that they may be separated by centrifugal force; the wax and wax-free oil are discharged continuously. T. A. S.

PETROLEUM—METHOD OF REFINING.—P. T. Sharples. U.S.P. 1,416,890 of May 23, 1922. (Appl. March 12, 1920.)

The residue, obtained after distilling off gas, naphtha and burning oils from crude petroleum, with the avoidance of substantial cracking, is chilled to precipitate the wax. The chilled residue is submitted to centrifugal treatment adapted so that the wax and wax-free residue are continuously discharged. T. A. S.

PETROLEUM OIL SLUDGES—SEPARATION OF.—S. H. Diggs. U.S.P. 1,418,781 of June 6, 1922. (Appl. Dec. 23, 1918.)

The acid sludge obtained in the treatment of petroleum oils with sulphuric acid is mixed with sludge obtained by treating oils with fuming acids and with gas oil. The whole is mixed with water and steamed when separation of the acid takes place. This is repeated until practically all the acid is removed. The residues are suitable for the manufacture of commercial asphalt. T. A. S.

HYDROCARBONS—PROCESS OF SEPARATING AND TOPPING—FROM A WATER MIXTURE.—W. A. Brown. U.S. Patent 1,419,610 of June 13, 1922. (Appl. Feb. 24, 1919.)

The process consists in heating the mixture under a pressure sufficient to prevent the vaporisation of the water, and thereby inducing the separation of the water from the oil. The hydrocarbons are then heated without heating the water, pressure being still maintained, and oil vapours are withdrawn. T. A. S.

CENTRIFUGAL OIL PURIFIER.—M. Leitch, Assr. to The De Laval Separator Company, New York. U.S.P. 1,415,881 of May 16, 1922. (Appl. April 22, 1920.)

A centrifugal machine having a compound rotatable bowl composed of two concentric members, of which the inner one alone has a perforated periphery, is furnished with means of rotating the latter, either independently, or in conjunction with the outer member. F. G. P. R.

PETROLEUM—APPARATUS FOR THE CONTINUOUS REFINING OF.—C. A. Jouett. U.S.P. 1,415,876 of May 16, 1922. (Appl. Sept. 23, 1918.)

Preheated oil is sprayed into the top of a vertical cylinder, divided horizontally into a number of chambers, which is heated by means of a concentric flue attached to a furnace stack provided with adjustable ports opposite each chamber. Annular traps are provided at the bottom of each chamber to allow unvaporised oil to flow into the top of the subadjacent and hotter one. Vapour pipes lead from each chamber to superposed dephlegmators, provided with run-back pipes to the next lower chamber, and thence through a similar dephlegmator system packed with a solid filtering medium to condensers. Fully-refined products are thus produced continuously. F. G. P. R.

REMOVAL OF SULPHURIC ACIDS FROM OILS REFINED WITH SULPHURIC ACID.—Handelsvenootschap Onder De Firma Jan Dekker. Holl. P. 5953 of July 15, 1921.

By the addition of aldehydes compounds are formed with the sulphonic acids dissolved in mineral oils refined by means of sulphuric acid, which are easily removable and are subsequently readily split again into their original components. F. G. P. R.

PREPARATION OF PURE DISTILLATES OF MINERAL OIL.—De Batsafsche Petroleum-Maatschappij. Holl. P. 2693 of Nov. 15, 1918.

Pure distillates of crude petroleum oil, which can be sold without further purification are obtained by adding from 5–20% by weight of H_3BO_3 to the oil and distilling. It is preferable to first put the H_3BO_3 in the still, heat up and then run in the oil. F. G. P. R.

LUBRICATING OILS.

EFFECT OF PARAFFIN WAX ON THE VISCOSITY OF PETROLEUM OILS.—E. W. Dean and M. B. Cooke. *J. Ind. Eng. Chem.* 1922, **14**, 410.

The object of the experiments was to ascertain the effect produced on the viscosity of various grades of oils by increasing or decreasing the quantity of paraffin wax dissolved therein. The oils employed were kerosene, two distillates having boiling ranges 250–275° C. and 275–300° C., transformer oil, spindle oil, medium auto oil and Liberty aero oil—all except the two distillates being commercial products, and in all cases derived from Pennsylvania crude. Additions of paraffin wax varied from 1 to 10%, and in the case of the distillates the amounts abstracted were of about the same order. Viscosity measurements were made at 100, 130 and 210° F. With kerosene the viscosity progressively increases with additions of wax, whilst with the transformer, spindle, auto and aero oils there is a progressive decrease which becomes more pronounced in the order given. In the case of the distillates the changes were practically negligible. As a general rule the viscosity of any mixture was found to be intermediate between that of the paraffin and wax free oil, but where the viscosity of wax and oil were practically equal that of the mixture was slightly less than either. Paraffin wax, in fact, appears to act like a liquid oil of equal viscosity, and this effect persists even at temperatures below its melting point. F. G. P. R.

METHODS OF MEASURING THE PROPERTY OF OILINESS.—R. E. Wilson and D. P. Barnard. *J. Ind. Eng. Chem.* 1922, **14**, No. 8, 683–695.

Oiliness is defined as the property of lubricants by means of which one fluid gives lower coefficients of friction than another fluid of the same viscosity. Various means of measuring this property are described, including the Deely type of machine for measuring coefficients of friction between plane surfaces at low speeds, a refined and reproducible method of determining static coefficients of friction between partially lubricated metal surfaces, the measurement of the inter-facial energy between oil and mercury, the measurements of the electrical resistance and the rate of formation of adsorbed films on metal surfaces, and the clogging of fine metal capillaries through which lubricants are forced. As a result of experiments it is believed that the static friction test is the best single test of the property of oiliness, but that it should be supplemented by measurements of the thickness of the adsorbed films at high pressures in order to obtain better knowledge of the action of different constituents

of lubricating oils. Animal and vegetable oils are almost invariably superior in oiliness to straight mineral oils, which can be greatly improved by admixture with these neutral glycerides. The addition, however, of small quantities of fatty acids or oil soluble soaps produces identical results. These experiments confirm the usual hypothesis that oiliness is due to selective adsorption of constituents in the oil by the metal surfaces, but make it incorrect to regard as monomolecular the adsorbed film, which acts merely by masking the attractive forces of the metal surfaces for one another. It is shown to be of colloidal dimensions, to be a plastic solid rather than a fluid film, and apparently acts by smoothing over surface irregularities, carrying much of the load and minimising metal to metal contact. The structure and physical characteristics of this film appear more important than its thickness in lowering the friction. By means of repeated treatment with finely-divided metals, such as iron-by-hydrogen, the constituents of lubricants which form these adsorbed layers may be selectively adsorbed and largely removed from the oil. F. G. P. R.

Oth—Process of Purifying and Dehydrating Transformer.—C. H. Howgood, Assr. to the De Laval Separator Co., New York. U.S.P. 1,425,645 of Aug. 15, 1922. (Appl. May 3, 1921.)

Transformer and similar oils containing only a very small proportion of water are practically completely dehydrated by being subjected to centrifugal force, whereby the water is carried to the periphery of the machine and there held as a surrounding film. The water-free oil floats on the watery envelope thus formed and is drawn off at a point substantially closer to the axis of rotation than the inner wall of the water film. F. G. P. R.

Transmission and Motor Oil.—W. K. Boileau, Assr. to Pittsburg Oil Refining Corporation. U.S.P. 1,423,512 of July 25, 1922. (Appl. June 5, 1922.)

The oil for the lubrication of cylinders, bearings and transmission of internal-combustion engines, and the fabric lining of transmission and brake parts, consists of petroleum oil of the ordinary consistency used in the cylinders of internal-combustion engines combined with 2–12% of neutral wool-fat. T. A. S.

Estimation of the Sludge Value of Transformer and Turbine Oils.—F. Schwarz and J. Marcusson. *Petroleum*, 1922, **18**, 741–742.

50 g. of oil are heated to 120° C. for 50 hours in a 200 c.c. Jena glass Erlenmeyer flask, of 7·5 cm. diameter, 12·5 cm. height, and about 25 mm. aperture of neck, by an electric heating element controlled by a thermostat. The oil is then heated for 15 min. to 80° C., under a dephlegmator with a mixture of 50 c.c. each of 50% alcohol (by weight), and a 4% solution of caustic soda, and afterwards strongly agitated for 5 min. The fluid mixture is then allowed to stand over night in a separating funnel. As much as possible of the mixture is filtered into a measuring cylinder and extracted with 30 c.c. light petroleum spirit. The resulting extract is treated with 10 c.c. of 50% alcohol, with the addition of a few drops of caustic soda. The alcoholic layer is mixed with the tar soap solution, and acidified with dilute hydrochloric acid. After shaking up two or three times successively, with 50 c.c. of benzene, the benzene extract is washed with distilled water till free from mineral acid, evaporated and dried at 105° C. From the amount of tarry matter obtained from the amount of fluid mixture taken its proportion to the oil employed may be calculated. H. M.

PREPARATION OF LUBRICANTS FROM LIGNITE TAR OIL.—M. Jacobsohn. *Brennstoff Chem.* 1922, **3**, 10–11.

In order to free the oil from objectionable phenolic bodies esterification is brought about by heating at 140° C. under a reflux condenser with ethyl alcohol in the presence of 10% of β–$C_{10}H_7HSO_3$ and washing the product with water. Oils containing up to 15% of phenols can be neutralised in this manner. F. G. P. R.

THE "FUSION" PATENT ROTARY RETORT.—C. J. Goodwin. Paper read before the South Wales Institute of Engineers, April 20, 1922.

The retort is designed for the recovery of oils from coal, shale, torbanite, etc. It consists of an inner tube about 18 in. in diameter, in which is situated eccentrically a four- or six-winged breaker or scraper, which is partially carried round by the rotation of the tube and then falls back, striking a glancing blow, and so keeping the heated surfaces free from scale, breaking up the material and preventing caking, the formation of carbon, and cracking. This tube is surrounded by an outer rotating tube about 2 ft. 6 in. in diameter, which is directly heated by the combustion of gases in a chamber underneath the tube. The material to be operated upon is fed into the inner tube, passes through the length of that tube, and returns through the outer tube and is discharged. The vapours evolved pass the length of the outer tube, and return through the inner tube, and escape by an outlet at the end of the latter. A rubbing joint made of flexible steel and asbestos prevents the leakage of vapours at the junction of the two tubes. The coke produced is in a finely-divided state, and would require briquetting for most purposes, but might find application in firing with powdered fuel. In trials 73·6 galls. of oil of 0·921 sp. gr. were obtained from a ton of Esthonian shale, the residue having 10·6% carbon content. Nova Scotia torbanite yielded 51·8, Australian torbanite, 131·7, and Kimmeridge shale 52·5 galls. per ton. Cracking was practically absent. H. M.

THE CHEMISTRY OF THE COKING PROCESSES. THE PRODUCTION OF BITUMINOUS SUBSTANCES OF HIGH-MELTING POINT, AND THEIR APPLICATION TO THE PRODUCTION OF METALLURGICAL COKE FROM NON-CAKING COAL.—F. Lierg. *Z. angew. Chem.* 1922, **35**, 264–268.

Coal tar pitch coked by itself gives a hard smooth coke. Sinter coal requires from 20% to 30% of pitch to make a good smooth coke, but if the pitch be dissolved in tar oil, 7%–9% suffices. This coking effect was not obtained in large scale working owing to the long period required for coking and the low melting point of the pitch. The coking effect is best attained by the use of bitumen of about 400° C. melting point. For the production of a bituminous extract of high melting point finely-powdered caking coal was exhausted for several hours with hot pyridine in an adaptation of the Soxhlet apparatus. By mixing the solution with water a precipitate was obtained, which dried to a brown powder, amounting to 20·9% of the coal used. Heated in carbon dioxide, the substance did not melt, but gave a swollen coke. The residuum of the extraction had lost all caking properties. The re-addition of the extract to the extracted substance did not restore its coking properties, but when the extract was dissolved in a solvent and added, the coking property was restored. A similar extract coking at 300° C. was made from coal tar pitch by extracting with benzol and xylol and evaporating. By further boiling with anthracene oil a seeming pitch-carbon, unaffected by a red heat, was obtained, but this gave off gas and yielded a hard coke

when heated in a crucible, showing it to be a mixture of organic compounds of high molecular weight. Illustrations show the behaviour as regards swelling in coking of these substances, and samples of various coals. It was demonstrated that coal melts on reaching coking point. The extracts of high melting point, on being mixed with various proportions of an inert substance (soot), still melted at the same point, but showed reduced degrees of swelling. The extract was somewhat soluble in hot pyridine, but deposited the dissolved portion again on cooling. Anthracene oil and heavier oils dissolved it almost entirely, and retained it in solution on cooling. The addition of extract in solution enables a hard coke to be made from brown coal. H. M.

Stoppage of Condenser in Distillation of Coal Tar.—W. Spaltenholz. *Chem. Zeit.* 1922, **46**, 544.

In distilling a tar with large water content, after the water and light oil had gone over, as also the first part of the creosote oil and most of the naphthalene, at about 300° C., the flow of oil from the condenser diminished. Crust-like formations resembling naphthalene were noticed at the condenser outlet. The flow of oil almost entirely stopped, and there followed an explosive ejection of hot oil mixed with a solid substance. The solid was found to be ammonium chloride, was fibrous and crystalline, and appeared to be produced to the amount of 5–10 kilograms per still. It was supposed to originate from coal which had been in contact with sea water during transport. H. M.

The Isolation of the Organic Substance in Esthonian Oil Shale.—J. Narbutt. *Z. angew. Chem.* 1922, **35**, 238–239.

The shale consisted approximately of one part of organic substance to two parts inorganic substance, and was of a light gray-brown colour. The finely-pulverised shale was treated with hydrochloric acid, and the mixture dried on a water bath. The solid, sticky mass was extracted with methyl alcohol in a Soxhlet apparatus till no reaction was obtained, with ammonium sulphide. The dark brown residue, after drying, was evaporated several times on water bath with hydrofluoric acid, and then with hydrochloric acid to turn the fluorides into chlorides. The mixture was washed with hot water with a slight addition of hydrochloric acid, and afterwards extracted with methyl alcohol in Soxhlet, till no reaction was given with ammonium sulphide. The substance obtained by this treatment is a dark brown powder heavier than water. On igniting, 2½% of ash, containing iron oxide, is left. On heating the organic substance to 330° C. with aromatic hydrocarbons of high molecular weight, such as naphthalene and phenanthrene, a partial solution took place, followed by the evolution of hydrocarbons and the formation of a nearly black solution. By heating the organic substance alone to 300°–350° C. a light brown oil, mobile at ordinary temperature, was obtained, and a black carbonaceous residue, while gas containing hydrogen sulphide was freely given off. H. M.

Recent Processes for the Exploitation of Oil Shale.—A. E. von Groeling. *Petroleum*, 1922, **18**, 487–493.

The varieties of oil shale are less capable of being standardised than are either petroleum or coal. The selection of the best apparatus for shale distillation depends on the nature of the shale, and also on the very different markets for products in different countries. The Scottish method aims at the production of ammonia, while the German manufac-

turer is more concerned with the yield of oil. The varieties of retort used in Scotland and their modes of working are described in detail. The Pumpherston retorts are not capable of dealing satisfactorily with some kinds of shale, for example, Brazilian. A form of retort is described, consisting of two concentric cylinders enclosing a cylindrical space, which served for the abstraction of the gases. The system worked under a vacuum of 27 inches of mercury, and gave from a ton of Scottish shale, 48·9 gall. crude oil, 8·9 gall. dry tar, and 33 lbs. of ammonium sulphate. The process was intermittent, requiring 2½–3 hours per charge of 450 lb. Unfortunately the high labour charges of the operation of this system rendered it unable to compete with the continuous retorts.

PART II.—*Petroleum* 1922, **18**, 539–545.

The vacuum retorts described give large yields of oil, and little permanent gas, owing to the quick removal of the products of distillation, and the good conduction of heat to the material to be distilled owing to the construction of the retort. Good results might be gained by preserving the principle of continuous operation in these retorts, while dropping the ring construction as being liable to cause difficulties in continuous working. American practice makes use of a modification of this process known as the "Digestion process." In this process the shale passes through a series of pulverisers, and is mixed with a stream of preheated heavy oil. It then passes through a mixing cylinder and into a still, named the digester, where it undergoes the first stage of the distillation process in a series of four horizontal cylindrical or rectangular chambers, from the lowest of which it is transferred successively to the upper chambers by a screw mechanism. Thence it is transferred to a separate chamber to undergo the final stage of the distillation. The firing is direct, and the combustion gases circulate contrary to the movement of the shale. The products of distillation are drawn by a slight vacuum through the chambers and pass into a preheater for the digestion oil. The shale from the first operation has the oil extracted from it by a centrifuge before undergoing the final stage of distillation. Colorado shale gave a yield of 87 Am. gall. oil per ton, in place of 51 gall. in Scottish retorts. The oils obtained by the first and the final distillations are respectively of 0·807 and 0·798 sp. gr., and have a content of unsaturated hydrocarbons of 24%. No permanent gases are evolved. The temperature of the first stage is 650° F., and of the final stage 1500° F. A yield of 30% of paraffin wax has been obtained from the oil produced in the distillation of certain kinds of shale. It is suggested that the results might be still further improved by the use of superheated steam.

H. M.

THERMOTECHNICAL RESEARCHES ON A BENZOL PLANT.—F. Plenz, K. Bode, and T. Werner. *Gas u. Wasserfach* 1922, **65**, 433–435.

The extraction of benzene from gas works and coke oven gas by washing with high-boiling point tar oil, and the recovery of the benzene by distillation with steam are purely physical processes, there being, as far as is known, no intermediate chemical reactions. The expenditure of energy in the washing process is comparatively small, but the benzene recovery requires considerable energy. The present investigation concerns the distillation process, exclusive of pumps, etc. The washing is a continuous process, whereas the recovery is intermittent. In the recovery the oil from the washing passes through a counter-current preheater, warmed by the exhausted washing oil, and through a steam

heater, whence it enters the distillation chamber. It then passes through the preheater and two coolers and back to the scrubber again. The distillation is effected intermittently with direct and indirect steam, and the benzene is condensed in a water-cooled condenser. Observations showed that the weight of steam used in distillation per unit of oil varied from 2·86 to 4·85, and of cooling water from 38·0 to 134·8, and 1·44 litres of washing oil are required for each cub. m. of gas. A diagrammatic representation of the heat distribution in the plant is given, and tables showing the heat transfer in the different members of the system.—(*To be continued.*) H. M.

Oil Shales and Coals.

Tar Distillation—Improvements Relating to.—Thermal, Industrial and Chemical Research Co., Ltd. J. S. Morgan and D. Rider. E.P. 184,624 of Aug. 24, 1922. (Appl. 15,726-21.)

The difficulties of frothing in tar-stills and the removal of lower boiling constituents from viscous pitch are overcome by feeding the crude preheated tar under the surface of molten metal, which is kept at such a temperature that hard pitch is discharged. The vapour from this still, which is kept about 350° C. is passed through a small fractionator, kept by the incoming tar at about 120° C. The vapours leaving at this temperature consist of water and light oils. The condensate from the fractionator is led into a secondary still, also containing molten metal, and maintained at about 230° C. This removes the usual tar fraction boiling at 170–230° C. The residues pass on to a third still kept at 270° C. where the fraction boiling at 230–270° C. is removed. The residue constitutes another well-marked fraction boiling between 270° C. and 350° C. A modification of the process consists in keeping the first still at 275–290° C., whereby the anthracene remains in the pitch. The pitch is then sprayed in a tower with superheated steam, which removes the anthracene. T. A. S.

Retorts for the Distillation of Oil-Bearing Shales, or other like Materials—Improvements in.—J. S. Black. Eng. Pat. 179,964 of July 29, 1921. (Appl. 30,724-20.)

The apparatus at the base of a vertical retort for the discharge of the spent shale and ashes is improved by supporting the contents of the retort on a platform, and having above this an angularly-oscillating member for the breaking up and discharge of the spent shale. The products of distillation are drawn off at various heights in the sides of the retort with a view to effecting a separation of the distillates. T. A. S.

Distillation of Tars or Oils—Process and Apparatus for.—E. Blumner. E.P. 182,868. (Appl. 936[1-21.)

The avoidance of direct contact between the oil and hot still walls is brought about by spraying the oil upwards through molten metal—*e.g.*, lead. T. A. S.

Continuous Distillation—Improved Stills for.—J. A. Yeadon. E.P. 183,682 of Aug. 3, 1922. (Appl. 17,775-21.)

A series of shallow oblong stills are used, through which the coal-tar or oil flows in succession by gravity. The bottoms of the stills are corrugated transversely, adding to the strength and heating surface of the stills. The stills are set at a slight inclination and oil flows from one

still to the next by means of a U tube so that the passage of vapour is prevented. Each still is fitted with its own condenser. T. A. S.

TAR—PROCESS AND APPARATUS FOR DISTILLING . . . AND OTHER LIQUIDS.—W. Glossop, L. Bradley, and R. P. Willsdon. E.P. 184,242 of May 2, 1921. (Appl. 12,524-21.)

The interior of a still is provided with superimposed plates, inclined at an angle to one another so that liquid delivered on to the top plate runs down and is delivered on to the higher part of the next plate, which is inclined in the reverse direction. In this way the liquid is caused to follow a zig-zag course in the form of a thin film, and when falling from one plate to the next produces a curtain of liquid, through which ascending vapours must pass. By this means the latter are well scrubbed, and also bring about a vaporisation of the light constituents in the liquid curtain. F. G. P. R.

HEAVY OIL—USE IN GAS MAKING.—O. B. Evans and H. G. Terziam. U.S.P. 1,418,782 of June 6, 1922. (Appl. July 2, 1920.)

The gas oil is gasified by introducing it to preheated checkerbrick. A deposit of carbon formed on the checkerbrick is removed by blasting it with previously heated air. T. A. S.

SHALE—PROCESS OF DISTILLING.—E. E. Hedges. U.S.P. 1,423,716 of July 25, 1922. (Appl. June 13, 1921.)

Oil-bearing shale is distilled in an elongated retort with constant agitation. The shale is moved along the retort, which is heated at one end to a temperature necessary to drive off the most volatile constituents, and at the other end to a temperature sufficiently high to decompose water in the presence of carbon. The discharged gases pass backwards through the progressively cooler portions of the retort. Steam is admitted at the hotter end of the retort from which the spent shale is removed. T. A. S.

OIL SEPARATOR.—J. C. Pool. U.S.P. 1,418,970 of June 6, 1922. (Appl. Feb. 18, 1920.)

The apparatus is for distilling oil from rock and consists of a long vertical tube divided into preheating, distilling and cooling sections. Arrangements are provided for drawing off the volatile oils from the distilling sections, for conveying heat from the distilling and cooling sections to the preheating section, and for the discharge of the residue from the bottom of the tubes. T. A. S.

RETORT FOR OIL SHALES AND SANDS.—C. L. Buckingham. U.S.P. 1,425,074 of Aug. 8, 1922.

A horizontal retort of U-shaped cross-section is provided along its sides with longitudinal flanges, which rest on brickwork walls composing the flue. A cover to the retort is provided with a plurality of gas domes, with outlet pipes to condensers. The shale or sand is fed in at one end of the retort and continuously moved along to the other end by suitable means, such as a screw conveyor. F. G. P. R.